Agilent G1888 Network Headspace Sampler

User Information

Agilent Technologies

Notices

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Safety Notices

CAUTION

A **CAUTION** notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in damage to the product or loss of important data. Do not proceed beyond a **CAUTION** notice until the indicated conditions are fully understood and met.

WARNING

A WARNING notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in personal injury or death. Do not proceed beyond a WARNING notice until the indicated conditions are fully understood and met.

Manual Overview

This manual contains 10 chapters and an index.

1 Introduction

Provides the purpose of this manual and an overview of its contents. It identifies the intended reader and lists the conventions used throughout the manual.

2 Introduction to Headspace and to the G1888 Network Headspace Sampler

Provides an overview of headspace theory and practice. It describes the instrument and its modes of operation.

3 The Keypad

Describes the keypad, the functions each key controls, and how to enter new setpoints for each function.

4 Advanced Functions

Describes the advanced functions controlled by the Adv Fctn menu, how to edit setpoints for these functions, and the displays that accompany each function.

5 Preparing a Sample

Describes the preparation of a sample for analysis, including filling and sealing the sample vial, loading the sample tray, and running a checkout sample.

6 Entering a Method

Describes the steps to follow in entering a method, including changing setpoints, storing and retrieving a method, and chaining methods.

7 Developing Methods

Describes the parameter changes that can affect sensitivity, precision, and accuracy.

8 Running a Sample

Summarizes operation of the Headspace Sampler to run a sample.

9 Maintaining the Instrument

Describes maintenance procedures including changing the sample loop, transfer line, needle, valves, and EPROM.

10 Troubleshooting and Error Messages

Lists the possible causes and solutions for chromatography problems or instrument failure.

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This chapter gives an overview of the manual and provides safety information.

About this Manual

The *G1888 Network Headspace Sampler User Information* manual explains how to use the G1888 Network Headspace Sampler. It includes all the basic information the user needs to develop methods, use the sampler to carry out analyses, and maintain and troubleshoot the sampler. For site preparation requirements and checkout procedures, see the G*1888 Network Headspace Sampler Site Preparation and Installation Manua*l.

Video supplements

Click this icon when it appears throughout the manual to view video supplements to certain procedures.

🎥

Intended User

This manual is intended for users who understand the basics of gas chromatography. Although the manual contains some headspace theory, it primarily provides operating, maintenance, and troubleshooting information for the G1888 Network Headspace Sampler.

This manual describes using the Headspace Sampler in standalone mode. However, many of the procedures covered in this manual also apply to users operating the Headspace Sampler with an Agilent integrated data system such as the Agilent Chemstation.

Conventions

The following conventions are used in this manual:

1. Braces { } enclose a generic name for which you should substitute specific information. For example, {setpoint} means you should key in the numeric value you desire for the setpoint.
2. Text in **Bold** means that you should press the key by that name. Be careful how you enter information. For example, the key sequence **Menu**, **.**, **1**, **Enter** indicates that you should press the **Menu** key, the **.** key, the **1** key, and then **Ente**r. It does not mean that you should press a comma (“,”). There is no comma on the sampler’s keypad.
3. Text in `Courier` indicates the message you will read on the sampler display.

Important Safety Warnings

Before moving on, there are several important safety notices that you should always keep in mind when using the Headspace Sampler.

Many internal parts of the instrument carry dangerous voltages

If the instrument is connected to a power source, even if the power switch is off, potentially dangerous voltages exist on:

- The wiring between the instrument power cord and the AC power supply, the AC power supply itself, and the wiring from the AC power supply to the power switch

With the power switch on, potentially dangerous voltages also exist on:

- All electronics boards in the instrument
- The internal wires and cables connected to these boards
- The wires for any heater (such as the oven)

WARNING **All these parts are shielded by covers. With the covers in place, it should be difficult to accidentally make contact with dangerous voltages. Unless specifically instructed to, never remove a cover unless the heated zones are turned off.**

WARNING **If the power cord insulation is frayed or worn, the cord must be replaced. Contact your Agilent service representative.**

Electrostatic discharge is a threat to instrument electronics

The printed circuit (PC) boards in the instrument can be damaged by electrostatic discharge. Do not touch any of the boards unless it is absolutely necessary. If you must handle them, wear a grounded wrist strap and take other antistatic precautions. Wear a grounded wrist strap any time you must remove the electronics cover.

Many parts are dangerously hot

Many parts of the instrument operate at temperatures high enough to cause serious burns. These parts include but are not limited to:

- The carousel and its contents
- The sample probe/loop assembly

You should always cool these areas of the instrument to room temperature before working on them. They will cool faster if you first set the temperature of the heated zone to room temperature. Turn the zone off after it has reached the setpoint. If you must perform maintenance on hot parts, use a wrench and wear gloves. Whenever possible, cool the part of the instrument that you will be maintaining before you begin working on it.

WARNING **Be careful when working behind the instrument. During cool-down cycles, the instrument emits hot exhaust which can cause burns.**

Gases

- Do not use flammable carrier gases.
- Do not use hydrogen as a carrier gas in the Headspace Sampler. Hydrogen creates a potential explosion hazard due to the venting of gases during operation and “standby”.
- Wear eye protection when using compressed gas to avoid eye injury.
- Fasten all compressed gas cylinders securely to an immovable structure or permanent wall.
- Store and handle compressed gases in accordance with relevant safety codes.
- Do not put gas cylinders in the path of a hot air vent (including a GC oven exhaust).

General warnings

- Perform periodic leak checks on supply lines, fittings, and pneumatic plumbing to prevent a potentially hazardous condition.
- To avoid a potential shock hazard when using liquid solution to locate leaks, turn the main power switch off and disconnect the main power cord. Be careful not to spill leak solution on electrical leads.

Safety and Regulatory Certifications

The Headspace Sampler conforms to the following safety standards:

- Canadian Standards Association (CSA): C22.2 No. 1010.1
- CSA/Nationally Recognized Test Laboratory (NRTL): UL 61010A–1
- International Electrotechnical Commission (IEC): 61010–1
- EuroNorm (EN): 61010–1

The instrument conforms to the following regulations on Electromagnetic Compatibility (EMC) and Radio Frequency Interference (RFI):

- CISPR 11/EN 55011: Group 1, Class A
- IEC/EN 61326
- AUS/NZ

This ISM device complies with Canadian ICES-001. Cet appareil ISM est conforme a la norme NMB—001 du Canada.

CE

The instrument is designed and manufactured under a quality system registered to ISO 9001.

Information

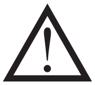
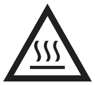
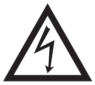
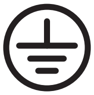
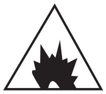
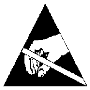
The Agilent Technologies Headspace Sampler meets the following IEC (International Electro-Technical Commission) classifications: Safety Class I, Transient Overvoltage Category II, Pollution Degree 2.

This unit has been designed and tested in accordance with recognized safety standards and is designed for use indoors. If the instrument is used in a manner not specified by the manufacturer, the protection provided by the instrument may be impaired. Whenever the safety protection of the Agilent Headspace Sampler has been compromised, disconnect the unit from all power sources and secure the unit against unintended operation.

Refer servicing to qualified service personnel. Substituting parts or performing any unauthorized modification to the instrument may result in a safety hazard.

Symbols

Warnings in the manual or on the instrument must be observed during all phases of operation, service, and repair of this instrument. Failure to comply with these precautions violates safety standards of design and the intended use of the instrument. Agilent Technologies assumes no liability for the customer’s failure to comply with these requirements.

See accompanying instructions for more information.	
Indicates a hot surface.	
Indicates hazardous voltages.	
Indicates earth (ground) terminal.	
Indicates explosion hazard.	
Indicates electrostatic discharge hazard.	

Electromagnetic compatibility

This device complies with the requirements of CISPR 11. Operation is subject to the following two conditions:

1. This device may not cause harmful interference.
2. This device must accept any interference received, including interference that may cause undesired operation.

If this equipment causes harmful interference to radio or television reception, which can be determined by turning the equipment off and on, the user is encouraged to try one or more of the following measures:

1. Relocate the radio or antenna.
2. Move the device away from the radio or television.
3. Plug the device into a different electrical outlet, so that the device and the radio or television are on separate electrical circuits.
4. Make sure that all peripheral devices are also certified.
5. Make sure that appropriate cables are used to connect the device to peripheral equipment.
6. Consult your equipment dealer, Agilent Technologies, or an experienced technician for assistance.
7. Changes or modifications not expressly approved by Agilent Technologies could void the user’s authority to operate the equipment.

Sound Emission Certification for Federal Republic of Germany

Sound pressure

Sound pressure Lp < 70 dB(A) according to DIN-EN 27779 (Type test).

Schalldruckpegel

Schalldruckpegel LP < 70 dB(A) nach DIN-EN 27779 (Typprufung).

Fuses

Table 1 lists the fuses required for proper operation. These fuses should only be accessed by Agilent service personnel.

Table 1 Fuses

Fuse designation	Location	Fuse rating and type
F2, F3	Power line module	10A 250V, glass tube
F3	Terminal near transformer	8A 250V, glass tube
F3, F4	Power board	6A 125V, glass tube
F1	Power board	1A 250V, glass tube

Cleaning

To clean the unit, disconnect the power and wipe down with a damp, lint-free cloth.

Recycling the Product

For recycling, contact your local Agilent sales office.

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This chapter briefly describes the theory and application of headspace analysis. It describes the parts of the G1888 Network Headspace Sampler and its modes of operation.

Overview of Headspace Analysis

Your new Headspace Sampler is a tool for introducing volatiles and some semi-volatiles that are contained in liquid or solid samples into a gas chromatograph. The analytical technique is called “static headspace gas chromatography.” The term “headspace” refers to the space above the liquid or solid sample that you place in a sealed vial. The term “static” means that the volatiles in the headspace of the vial are at equilibrium with the same compounds in the liquid or solid sample.

The instrument extracts a reproducible volume of the headspace and injects it into the carrier gas flowing into the gas chromatograph. Food, packaging, pharmaceutical products, soil, and water are often tested to see if they meet quality criteria or regulatory standards. Fragrances and consumer products are analyzed using headspace in order to understand their composition. Forensics laboratories use headspace for measuring the percent of ethanol in human blood to see if a driver is legally intoxicated.

The ease with which a sample forms a gaseous headspace depends on the sample’s “partition coefficient.” See “Developing Methods” on page 108 for a more thorough explanation. After a programmed heating time, the headspace gas is extracted from the vial and injected into a GC for analysis.

There are two means of extracting the headspace gas from the vial—single or Multiple Headspace Extraction (MHE). With single headspace extraction, only one sample of gas is taken from each sample vial. Single headspace extraction is the standard method. It is used for samples that are readily volatile or for which there are no matrix effects or limited matrix effects. Quantitative analysis can be performed by using suitable internal or external standards.

MHE is used for headspace samples for which no calibration standard can be prepared easily (for example, solid samples) or for highly volatile compounds in liquids, where the method of standard addition is difficult because of losses due to evaporation. MHE takes several headspace samples from each vial. With each extraction, the concentration of the volatile compound in both the gas phase and the sample becomes smaller, although the ratio remains constant. The logs of the peak areas versus the extraction number are plotted to obtain quantitative data about the compound of interest.

MHE is often used as a calibration tool. Once the peak area versus the concentration is established for a given sample matrix, quantitative measurements of samples with a similar matrix can be carried out using a single extraction.

The G1888 Network Headspace Sampler

The G1888 Network Headspace Sampler provides an automated method to run up to 70 samples consecutively without operator attention. The sampler's microprocessor optimizes the time spent for each run according to the values programmed in by the operator. Up to four different methods can be stored and used to analyze a series of samples.

The Headspace Sampler can also do multiple headspace extraction (MHE) using multiple septum punctures, with up to 100 extractions per sample. MHE can also be used to automate optimization routines.

A second method of MHE named the CONC mode is available. CONC mode allows sample concentration at the head of the column for trace analysis.

See Figure 1 and Figure 2 for a visual overview of the Headspace Sampler.

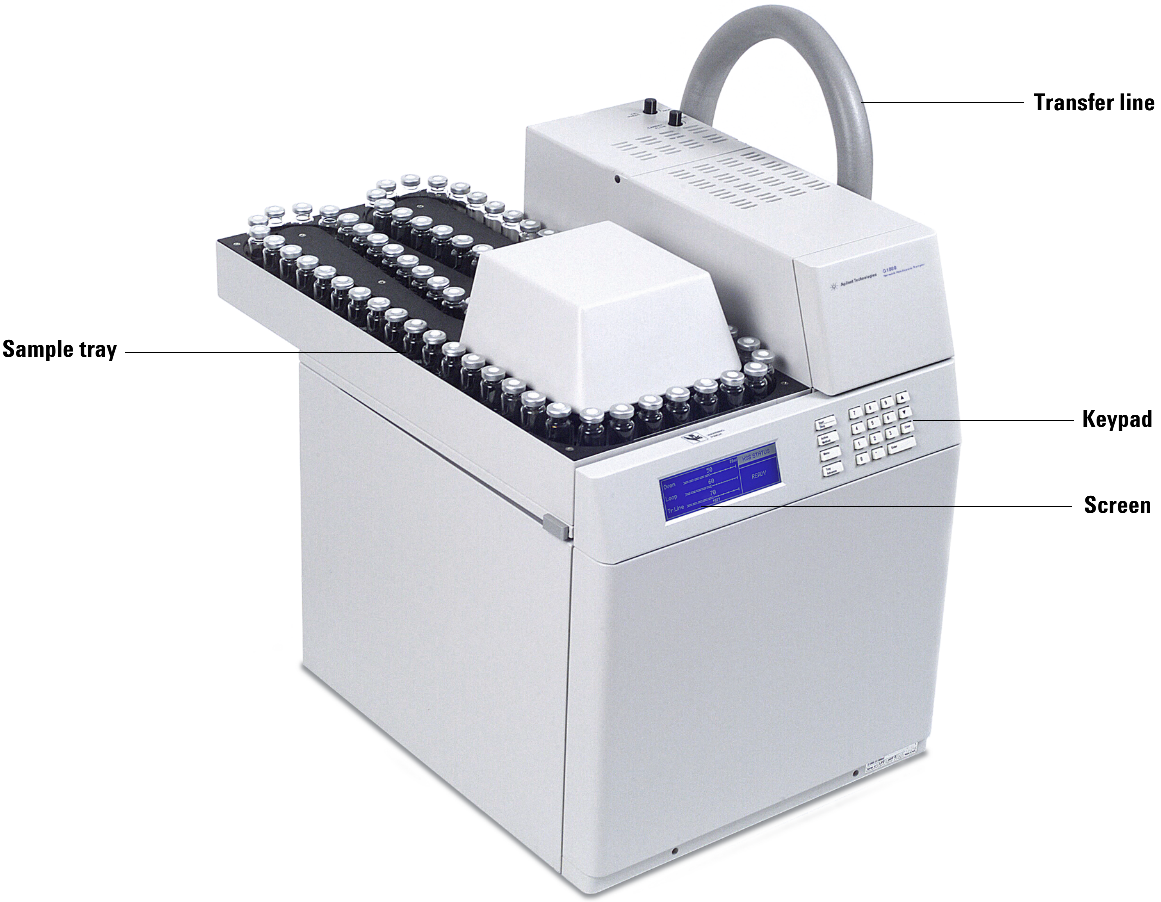

Transfer line
Sample tray
Keypad
Screen

Figure 1 Overview of the G1888 Network Headspace Sampler (from the front of the instrument)

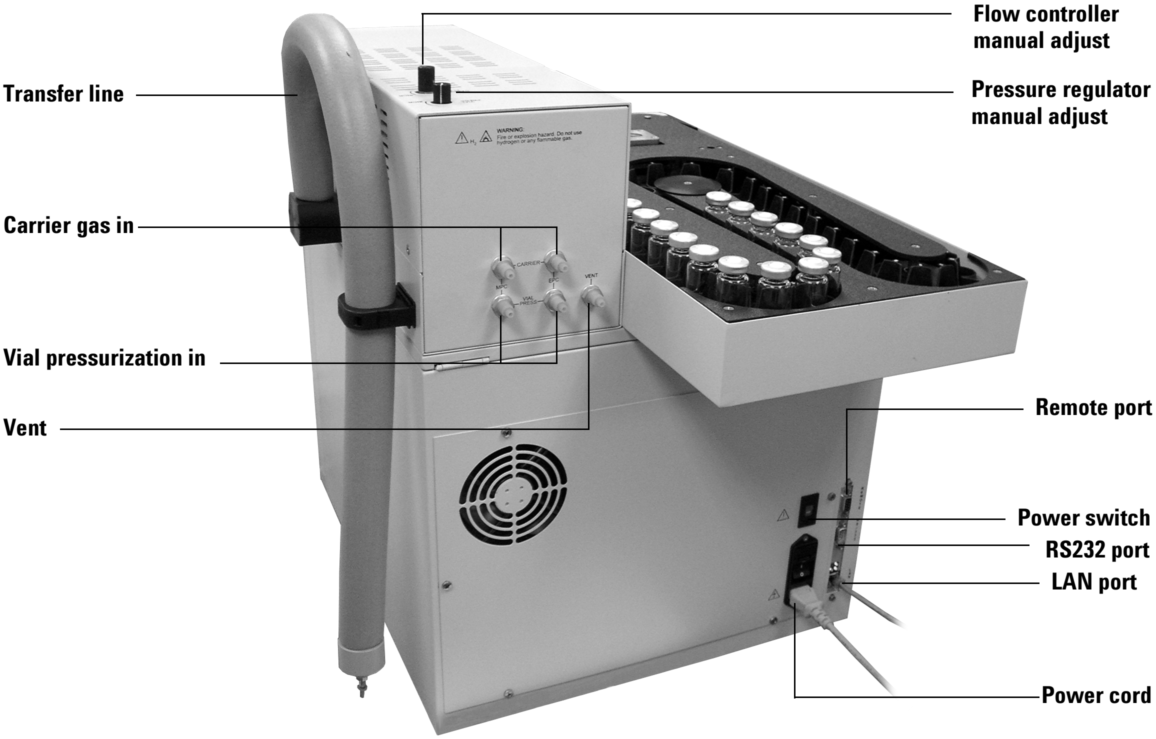

Flow controller manual adjust
Pressure regulator manual adjust
Transfer line
Carrier gas in
Vial pressurization in
Vent
Remote port
Power switch
RS232 port
LAN port
Power cord

Figure 2 Overview of the G1888 Network Headspace Sampler (from the back of the instrument)

The Headspace Sampler is made up of the following components:

Oven

The oven contains a circular aluminum sample carousel that holds up to twelve 10-mL or 20-mL sample vials. A resistance band heater heats the oven. A motor–driven fan in the center of the carousel circulates the air to maintain constant and uniform temperature. The carousel can shake at two speeds to help components elute into the headspace quicker. Mechanical rods move vials down into the oven and up to the needle for extraction.

Heated zones

There are three heated zones. They are the vial oven, the loop, and the transfer line. Each heated zone can be set to a specific temperature or turned off.

The vial oven is where the vials heat during thermal and chemical stabilization. The setpoint temperature range is between 40 °C and 230 °C. At this point, the needle has not pierced the septum.

The loop zone controls the temperature of the upper chamber, the valve, and the sample loop. The setpoint range of the loop is between 45 and 250 °C. Keep the loop temperature 10 to 15 °C higher than the vial temperature for most operations.

The temperature range of the transfer line is 50 to 250 °C.

Vial tray

The 70-sample vial tray is located on top of the unit to the left of the oven. The tray is constructed of a flexible belt with dividers for each vial compartment. The keypad on the front of the Headspace Sampler controls tray rotation to assist filling the tray with headspace vials. The tray can move forward and backward.

Sampling system

The basic components include a deactivated stainless steel vial–sampling probe with an inner diameter of 0.5 mm, six–port valve, deactivated stainless steel valve sample loop, set of two miniature solenoid valves, and deactivated stainless steel tubing.

Transfer line

This line connects the Headspace Sampler to the GC. The transfer line is 85 cm long and made of deactivated stainless steel. The transfer line heats to a maximum temperature of 250 °C. There is a removable needle for easy interfacing through GC inlet septa.

Keypad and display

The Headspace Sampler has a 19–key elastomeric keypad with a graphic LCD display. All functions of the sampler are controlled from the keypad with the exception of EPC, which is controlled from the GC or ChemStation.

Pneumatics

The Headspace Sampler uses two flow paths: Carrier gas flow and flow for vial pressurization. There is a manual flow controller for control of the GC carrier gas flow and a manual pressure regulator for control of the vial pressurization. Use the manual flow controller and manual pressure regulator when the Headspace Sampler is set for manual pressure control (MPC).

When the Headspace Sampler is set for electronic pressure control (EPC), the GC provides all flow and pressure control.

Toggle the flow mode between EPC and MPC using the switches under the lid (Figure 3). Carrier gas and vial pressurization gas do not have to be set to the same flow mode.

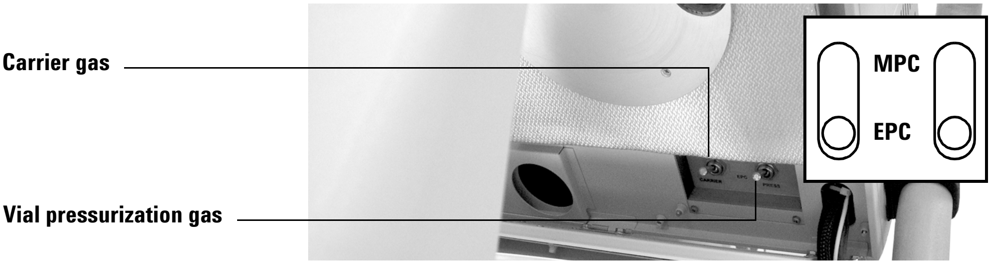


Figure 3 EPC/MPC toggle switches

Four 1/8–inch Swagelok® bulkhead fittings are provided at the rear of the pneumatic compartment for connecting vial pressure and carrier gas lines. Two fittings are for MPC, and two fittings are for EPC.

See Figure 4 and Figure 5 for diagrams of the pneumatics system. The system is split into two drawings for clarity.

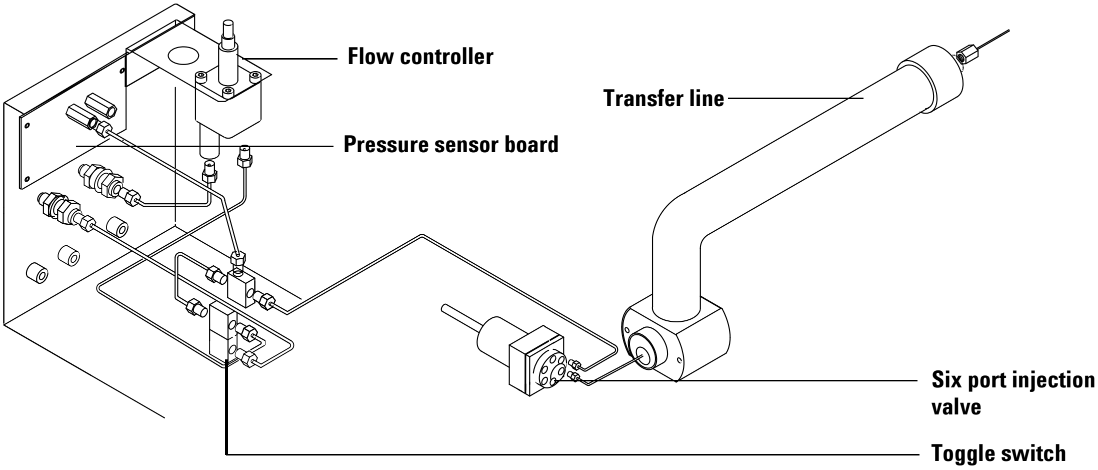

Flow controller
Transfer line
Pressure sensor board
Six port injection valve
Toggle switch

Figure 4 Carrier gas pneumatic system for the Headspace Sampler

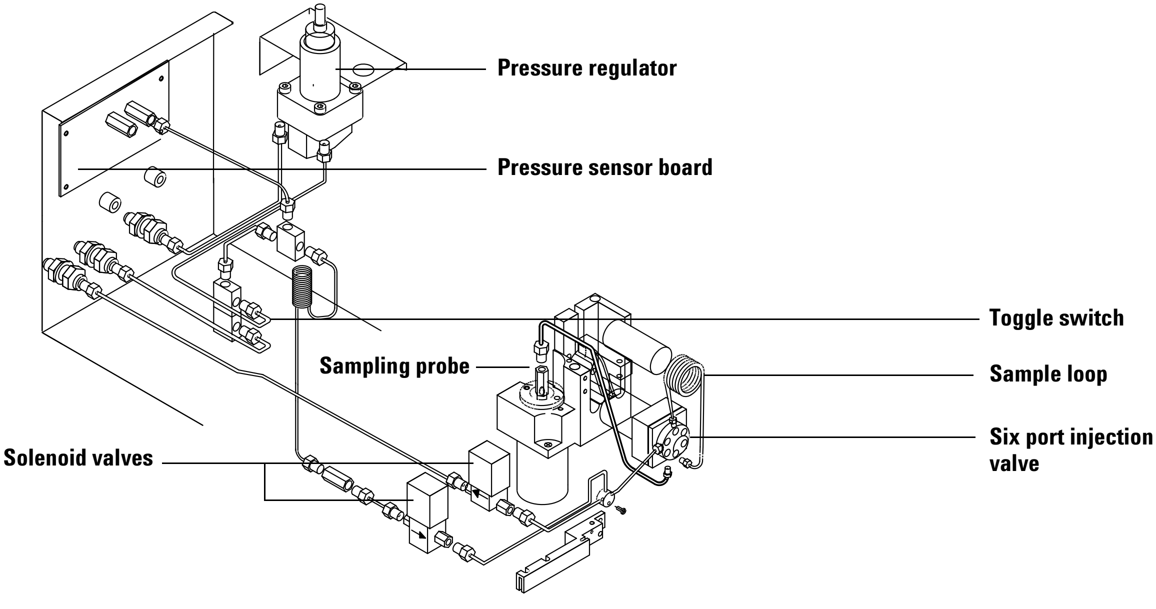

Pressure regulator
Pressure sensor board
Toggle switch
Sampling probe
Sample loop
Six port injection valve
Solenoid valves

Figure 5 Vial pressurization gas pneumatic system for the Headspace Sampler

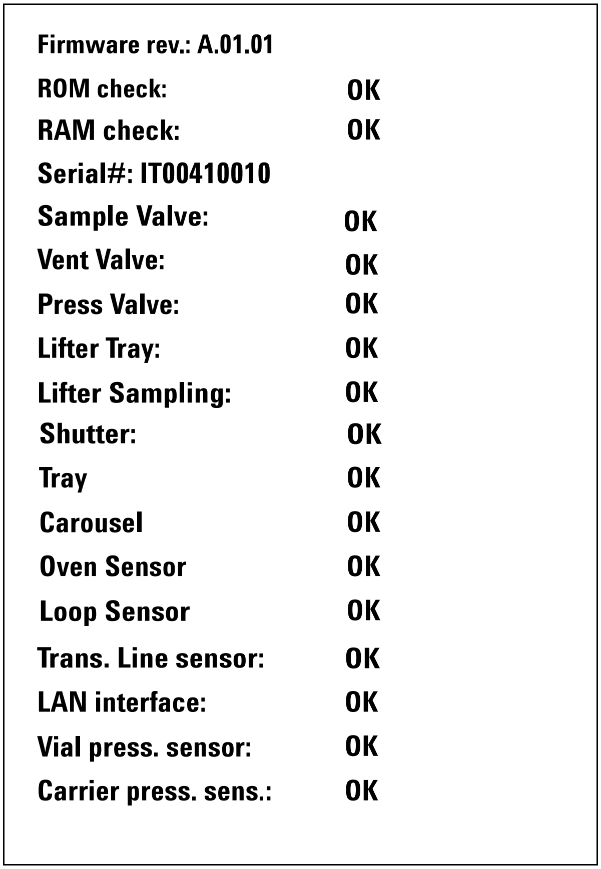
Principles of Operation

General operation

When the instrument is switched on, the sampler moves the first vial into position. The Headspace Sampler does not place the vial in the oven at this time. The oven, sample loop, and transfer line heat to reach the setpoint values. The display reads “Not Ready” until the oven, sample loop, and transfer line reach their setpoints. If you press Start/Stop before the machine is ready, the display reads “Waiting for Ready”. When setpoints are reached, the display changes to “Ready” and the Headspace Sampler places the first vial into the oven.

Power on diagnostics

When the instrument is powered on or reset (see page 78), it runs through a series of checks to make sure it is operating properly. Figure 6 shows the startup diagnostic screen.



```
Firmware rev.: A.01.01
ROM check:               OK
RAM check:               OK
Serial#: IT00410010
Sample Valve:            OK
Vent Valve:              OK
Press Valve:             OK
Lifter Tray:             OK
Lifter Sampling:         OK
Shutter:                 OK
Tray                     OK
Carousel                 OK
Oven Sensor              OK
Loop Sensor              OK
Trans. Line sensor:      OK
LAN interface:           OK
Vial press. sensor:      OK
Carrier press. sens.:    OK
```

Figure 6 Startup diagnostics

Other messages may appear as well. If you receive other messages, see Troubleshooting and Error Messages on page 163.

Standby Condition

During standby, the pressurization valve is open. Auxiliary gas flushes the sample valve, the sample loop, and the sampling needle. Carrier gas flows to the GC injection port through the sample valve. See Figure 7.

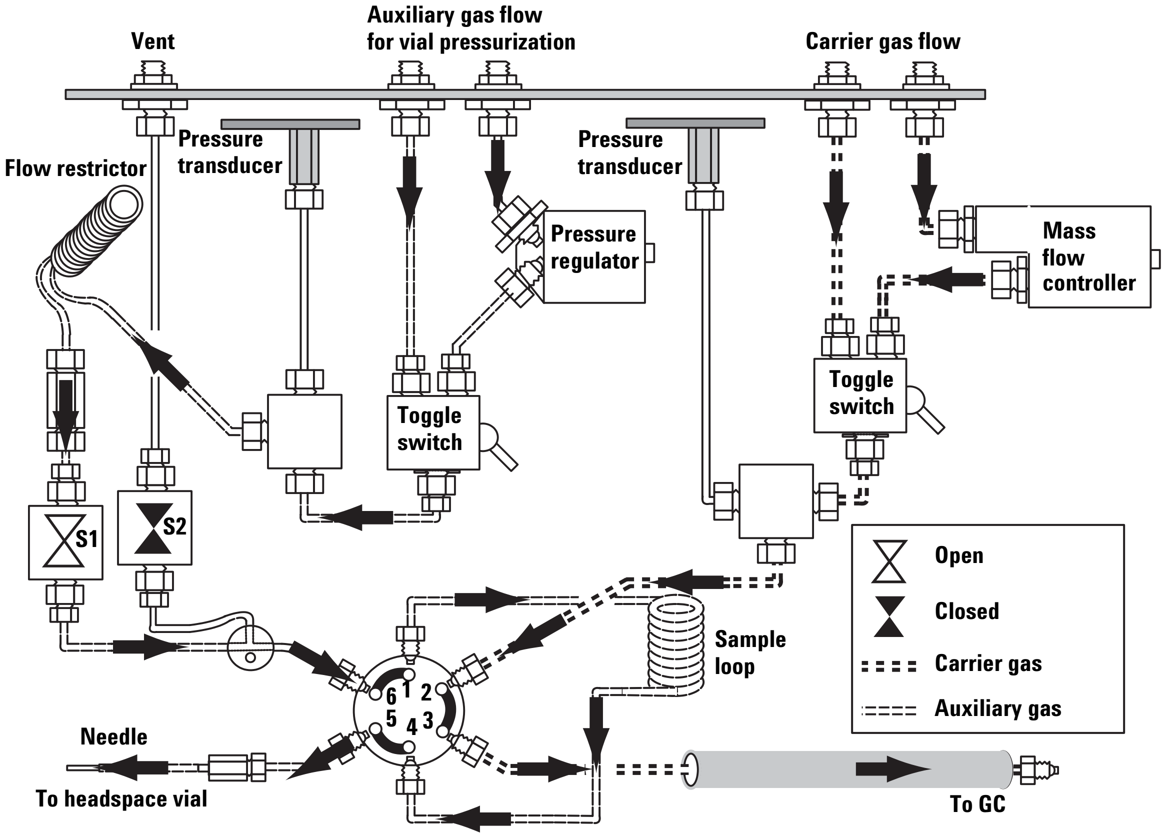

Vent
Auxiliary gas flow for vial pressurization
Carrier gas flow
Flow restrictor
Pressure transducer
Pressure transducer
Pressure regulator
Mass flow controller
Toggle switch
Toggle switch
S1
S2
Open
Closed
Carrier gas
Auxiliary gas
Sample loop
6
1
2
5
4
3
Needle
To headspace vial
To GC

Figure 7 Standby condition. S1 is open

Vial pressurization

At the end of the vial equilibration time agitation stops, the vial moves in-line with the sampling probe, and the mechanical rod raises the vial onto the sampling probe. The pressurization valve closes. The sampling probe pierces the septum of the vial. The pressurization valve opens, allowing gas to enter the vial for the pressurization time. The valve then closes again. See Figure 8.

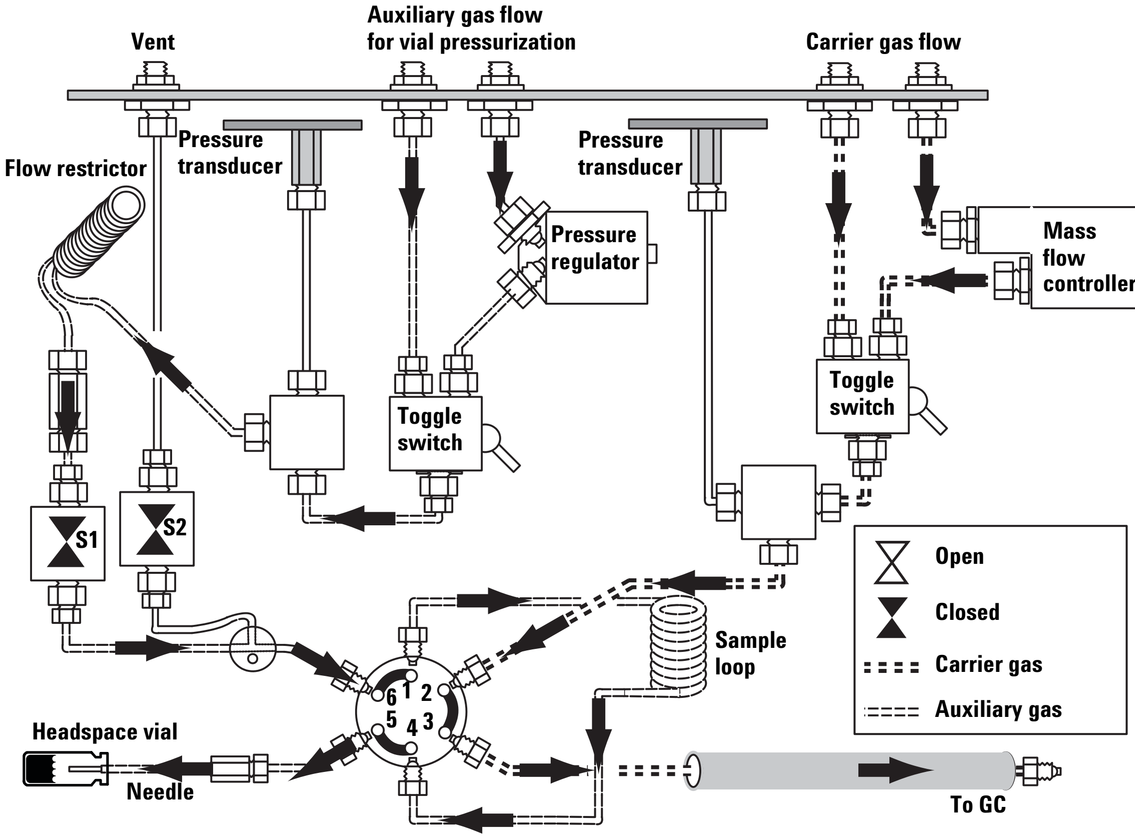

Vent
Auxiliary gas flow for vial pressurization
Carrier gas flow
Flow restrictor
Pressure transducer
Pressure transducer
Pressure regulator
Mass flow controller
Toggle switch
Toggle switch
S1
S2
6
1
2
5
4
3
Sample loop
Headspace vial
Needle
To GC
Open
Closed
Carrier gas
Auxiliary gas

Figure 8 Vial pressurization. S1 is closed (vial raised on needle).

Filling the sample loop

The vent valve opens and the headspace gas fills the sample loop. It then vents to atmospheric pressure through the vent outlet. A short loop–fill time (2 to 5 seconds) may leave pressure in the loop equilibrated to the vial pressure and allow more analyte to be injected. A longer loop–fill time (10 to 15 seconds) equilibrates the loop pressure to atmospheric pressure. See Figure 9.

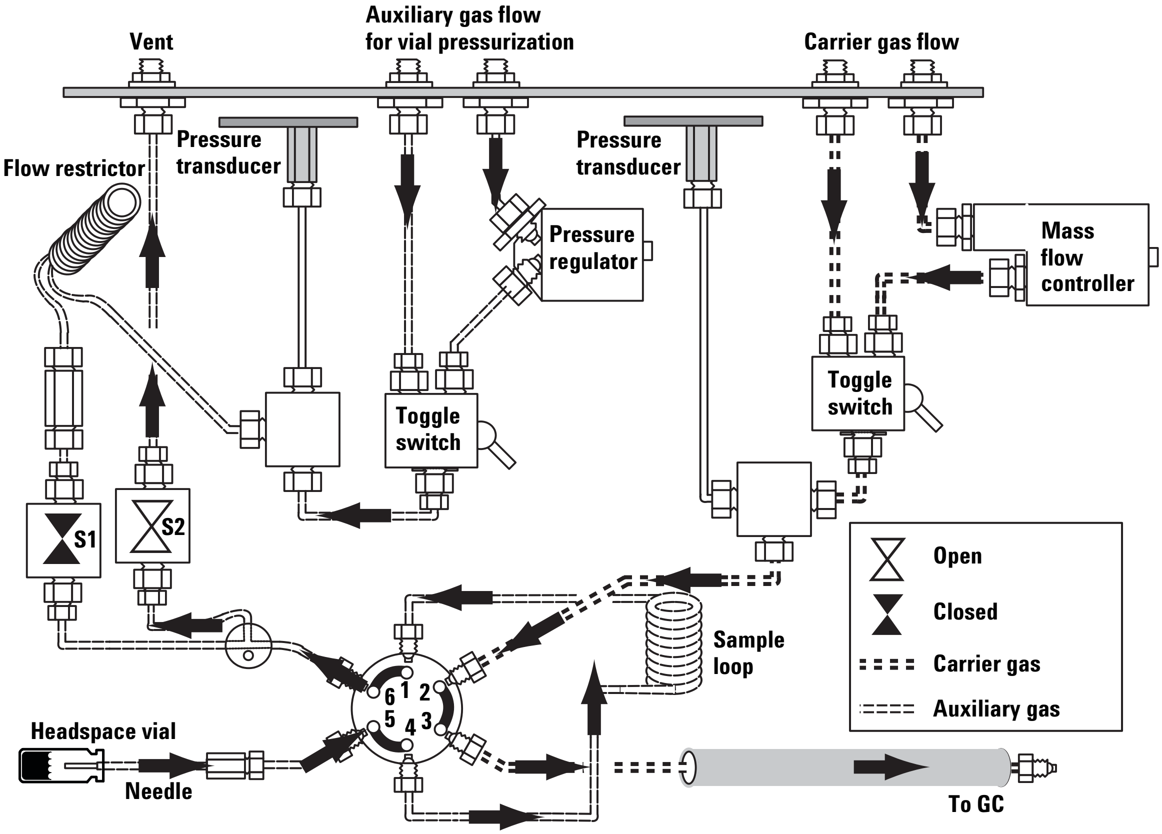

Vent
Auxiliary gas flow for vial pressurization
Carrier gas flow
Flow restrictor
Pressure transducer
Pressure transducer
Pressure regulator
Mass flow controller
Toggle switch
Toggle switch
S1
S2
6 1 2 5 4 3
Sample loop
Headspace vial
Needle
To GC
Open
Closed
Carrier gas
Auxiliary gas

Figure 9 Loop fill. S1 is closed. S2 is open to fill the loop.

Loop equilibration

Both pressure and vent valves are closed for the length of time assigned to this setpoint.

Injecting the sample

The sample valve brings the sample loop in line with the carrier gas flow. The carrier gas flows through the sample loop and the transfer line into the GC inlet port. This sweeps the sample into the GC. See Figure 10.

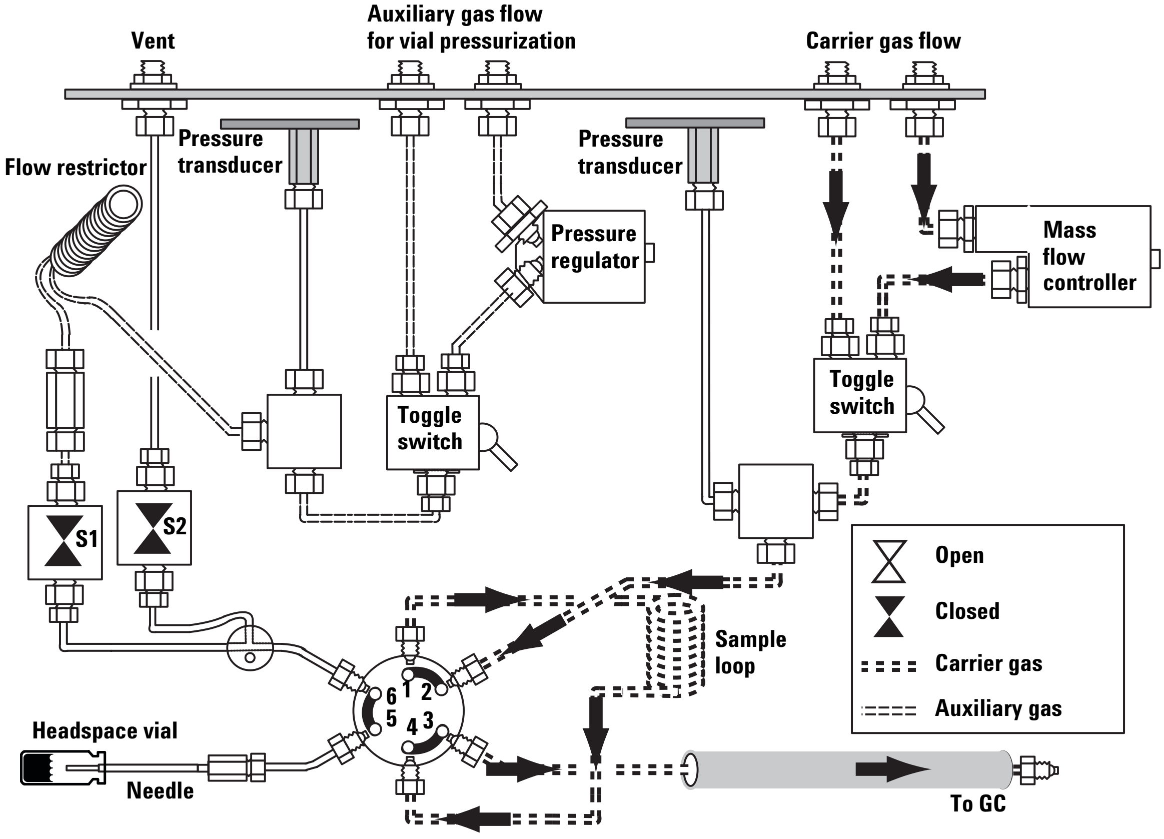

Vent
Auxiliary gas flow for vial pressurization
Carrier gas flow
Flow restrictor
Pressure transducer
Pressure transducer
Pressure regulator
Mass flow controller
Toggle switch
Toggle switch
S1
S2
Sample loop
6
1
2
5
4
3
Headspace vial
Needle
To GC
Open
Closed
Carrier gas
Auxiliary gas

Figure 10 Injection. S1 and S2 are closed.

End of cycle and vial recovery

The sample vial lowers into the carousel. The carousel rotates and the lift returns the vial to the tray. The vent valve opens to purge the vent line. The sampler returns to the standby configuration. See Figure 11. The default vent time for S2 is 30 seconds. To adjust the vent time, see Vent Valve purging on page 84.

.

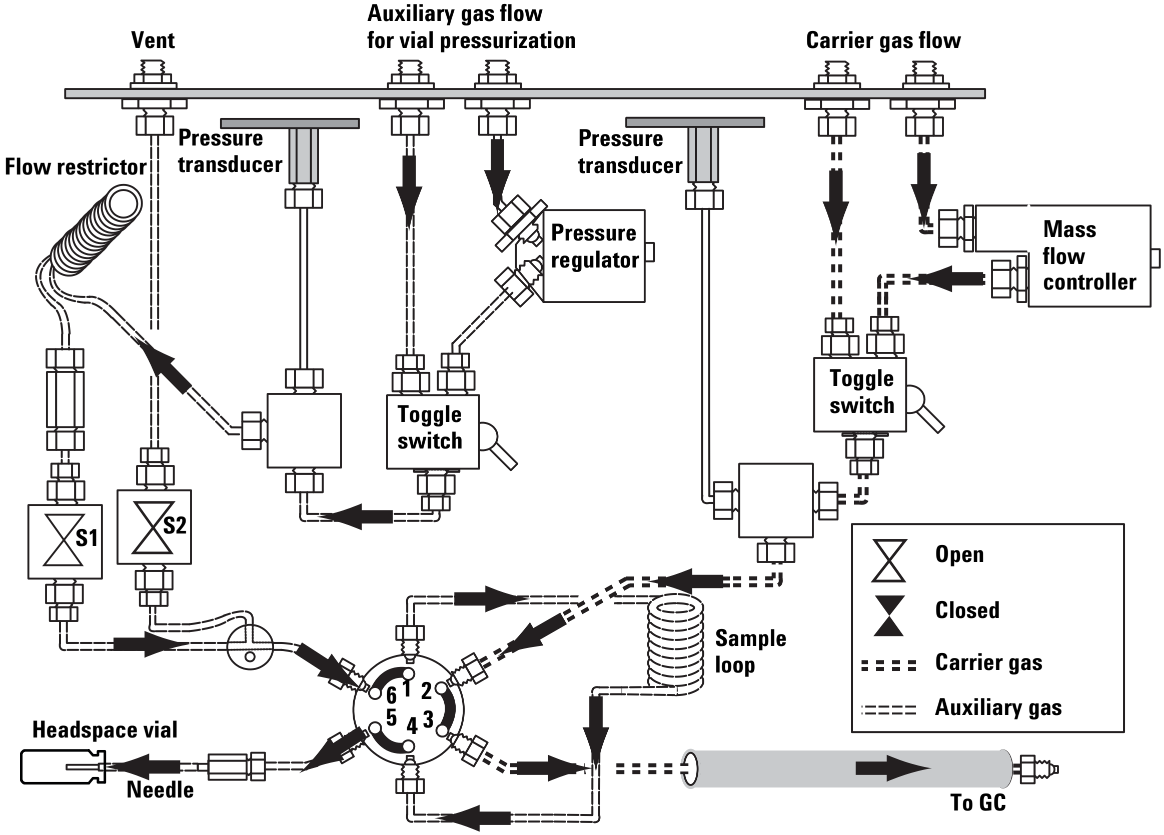

Vent
Auxiliary gas flow for vial pressurization
Carrier gas flow
Pressure transducer
Flow restrictor
Pressure transducer
Pressure regulator
Mass flow controller
Toggle switch
Toggle switch
S1
S2
Open
Closed
Carrier gas
Auxiliary gas
Sample loop
6
1
2
5
4
3
Headspace vial
Needle
To GC

Figure 11 Purge. S1 is open. S2 is open for 30 seconds.

Modes of Operation

The G1888 Network Headspace Sampler has two modes of operation—standard headspace extraction and multiple headspace extraction.

Standard headspace extraction

In standard headspace extraction mode, one extraction is made per vial. The GC run starts when the Headspace Sampler injects the sample into the GC inlet. In order to maintain the same incubation interval during the gas chromatographic run, the Headspace Sampler transfers vials one by one into the oven and recovers each vial after sampling. More than one vial can be present in the oven simultaneously. The sampler analyzes the event times and the GC cycle time, then adjusts vial delivery and recovery times to optimize overall process time. Vial agitation is allowed in this mode.

Note that the GC run cycle time is the time from injection until the system is ready for another injection. Occasionally, the time for data handling becomes a factor.

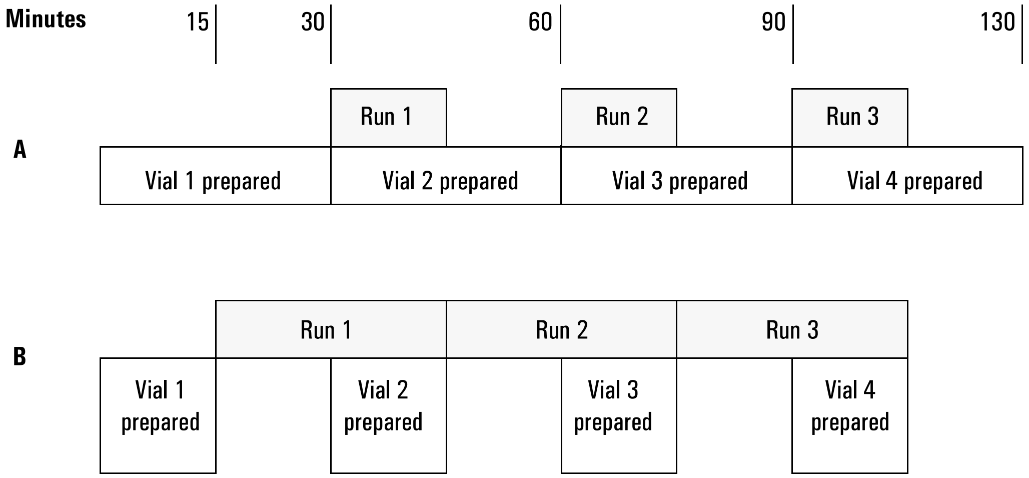

Minutes
15
30
60
90
130
A
Run 1
Run 2
Run 3
Vial 1 prepared
Vial 2 prepared
Vial 3 prepared
Vial 4 prepared
B
Run 1
Run 2
Run 3
Vial 1 prepared
Vial 2 prepared
Vial 3 prepared
Vial 4 prepared

Figure 12 Instrument synchronization

Figure 12 shows how the instrument synchronizes the availability of a vial which has been heated and is ready for injection with the GC’s readiness for another injection. For simplicity, the two cases are:

- Situation A: total preparation time of 30 minutes with a run cycle time of 15 minutes.
- Situation B: total preparation time of 15 minutes with a run cycle time of 30 minutes.

Sequence of events

The normal sequence of events is:

1. Standby condition. The Headspace Sampler is at the previous method setpoints.
2. Load method.
3. The system waits for the oven equilibration time.
4. Run/Method start. The first vial moves into oven.
5. If agitation (shaking) is set, the carousel shakes for a preset time.
6. The oven heats the vial for the vial equilibration time.
7. The vial fills with pressurization gas.
8. Vial sampled. Sample loop fills.
9. Sample loop equilibrates.
10. Headspace Sampler makes the headspace injection and triggers a GC start.
11. The vial returns to the carousel.
12. The vial returns to the tray

Multiple headspace extraction with multiple vial puncture

MHE mode can make up to 100 extractions per vial. With multiple vial puncture, the needle exits the vial and the vial returns to its original position in the oven after an extraction. Vial agitation is allowed.

MHE is not allowed if Parameter Increment (Advanced Function 2) is on.

In MHE, users can encounter two possible situations:

- GC Cycle Time is longer than the Vial Equilibration Time

  The first extraction begins after the Vial Equilibration Time is satisfied. Each subsequent extraction occurs based on the cycle time interval.

  For example, if Vial Eq. Time = 5.0 min and Cycle Time = 10.0 min, then first extraction is at 5.0 min, second is after another 10 min, (15 min cumulative, approx.), third after another 10 min (25 min. cumulative), etc.

- GC Cycle Time is shorter than the Vial Equilibration Time

  The first extraction begins after the Vial Equilibration Time elapses. Each subsequent extraction occurs based on the Vial Eq. time interval.

  For example, if Vial Eq. Time = 8.0 min and Cycle Time = 5.0 min, then first extraction is at 8.0 min, second is after another 8 min, (16 min cumulative), third after another 8 min (24 min. cumulative), etc.

The following events take place during MHE with multiple vial puncture:

1. Multiple extraction method loaded
2. Vial placed in oven
3. Vial heated for vial equilibration time
4. Vial sampled according to event times
5. GC started
6. Vial returned to slot in oven carousel
7. Second heating time started
8. Vial sampled according to event times
9. GC started
10. Vial returned to oven
11. Continue from step 7 (up to number of extractions per vial)
12. Vial returned to tray

Multiple Headspace Extraction: Standard

In this MHE mode, the Headspace Sampler samples each vial multiple times. The GC receives a start signal after each extraction from the vial. See Multiple Headspace Extraction on page 75 for more details. Usually, four extractions per vial are adequate to obtain a good log (area) versus extraction number graph for quantitative work if appropriate temperature and equilibration times are set.

Multiple Headspace Extraction: Conc mode

In this MHE mode, the Headspace Sampler samples a vial multiple times to concentrate the analytes at the head of the GC column or in a cryo trap at the GC inlet. The GC receives a start signal after the final extraction from the vial. See Multiple Headspace Extraction on page 75 for more details. This technique is useful for maximum sensitivity.

3

The Keypad

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  Editing setpoints 48
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  Enter 50
  Clear 50
  Numeric keypad 50
  Tray Advance 50
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  Chain Methods 61
  Pressures 62

This chapter describes each of the keys and the functions associated with the keys.

Accessing Functions

Use the keypad and screen on the front of the instrument to view and change all settings. Press **Menu**, **Active Method**, or **Tray Advance** once to bring up a menu of parameters connected with that key. Use the up and down cursor keys to scroll through the menu items available for control. The instrument display shows the current value of the setpoint for the highlighted parameter.

Editing setpoints

To edit a setpoint:

1. Navigate to the parameter.
2. Key in the new value using the numeric keypad.
3. Press **Enter** to make the new value part of the active method.

   If the new value is not within the allowed range, the sampler displays a message showing the allowed range. Press any non–number key to remove the error message.

At any point in a menu, press **Clear** to return to the top–level display.

See Table 2 for a quick reference of the keys and their functions.

The Keypad

Table 2 below lists all the keys on the Headspace Sampler, and summarizes their functions. See Keys and Their Functions on page 50 for details.

Figure 13 shows the layout of the keypad.

Table 2 Quick reference of keys and functions

Key	Definition/Function
Start/Stop	Starts, Stops and/or resets a run
Active Method	Displays a menu for editing method parameters
Menu	Displays the main menu for access to advanced functions and loading, storing, and chaining methods
Tray Advance	Moves tray one position
Cursor keys	Navigates through menu options
Clear	Cancels a keyboard entry. Displays menu top level
Enter	Adds keyboard entry to current method or executes highlighted selection

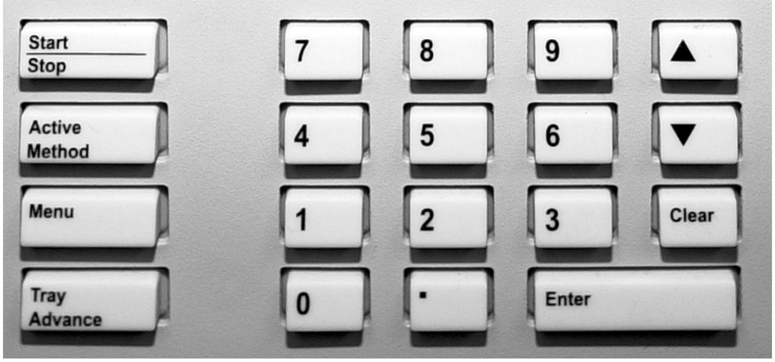


Figure 13 The keypad

Keys and Their Functions

Enter

This key is used after entering a numeric value for a setpoint. Press **Enter** to incorporate the change into the active method. The Enter key will also select a highlighted menu item.

Clear

Press the **Clear** key during editing to return the value to its previous setpoint. The Clear key also returns the display to a higher menu level. You may need to press Clear more than once to reach the top level.

Numeric keypad

The numbered keys are used to enter setpoint values for the variable conditions of the Headspace Sampler. The decimal key is used for menu shortcuts.

Tray Advance

Press this key to enter the Tray Advance screen and manually position the tray. Press the cursor keys while in the Tray Advance screen to move the tray 1 position in either direction. Type a number between 1 and 70 using the keypad followed by Enter to move directly to a specific tray position.

Press **Advance Tray**, {position+7}, and **Enter** to move the desired loading position to the very front of the sample tray. For example, enter 67 to bring tray position 60 to the front of the machine. Note that position 1 follows position 70. For example, enter 5 to bring position 68 to the front of the machine.

Start/Stop key

Press this key to start or stop a run. To start a run, press once. To stop a run, press the key again. The display reads:

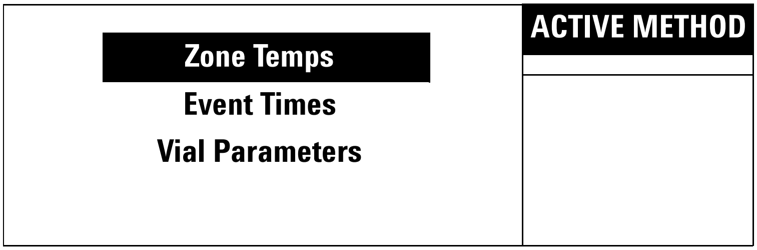
PRESS **STOP** TO CONFIRM

This prevents an inadvertent run interruption. To stop, press the key again. To continue the run, press **Clear**.

- When the sampler stops during a run, the vials in the oven automatically return to the sample tray. The method cannot be resumed. To continue the analysis from the last vial sampled, change the method so that it starts with the correct vial.

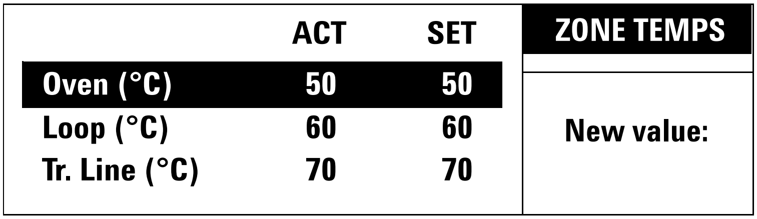
The Active Method Key

Press the Active Method key to display the following menu:


ACTIVE METHOD
Zone Temps
Event Times
Vial Parameters

- For more information on Zone Temps, see page 52.
- For more information on Event Times, see page 54.
- For more information on Vial Parameters, see page 57.

Zone Temps


ZONE TEMPS
New value:

	ACT	SET
Oven (°C)	50	50
Loop (°C)	60	60
Tr. Line (°C)	70	70

Description

Zone Temps sets temperature values for various heated zones in the sampler.

Procedure

Use the cursor keys to scroll to OVEN, LOOP or TR. LINE (Transfer Line) to set temperature values. Enter a new value using the numeric keypad. Press **Enter** to set the value. See Table 3 for the allowed temperature ranges.

Table 3 Zone temperature ranges

Heated zone	Allowed setpoint range	Default value (°C)
Oven	40–230 in 1 °C increments *	50
Loop	45–250 in 1 °C increments	60
Tr. Line	50–250 in 1 °C increments	70

*The minimum oven temperature is 40 °C or 10 °C above ambient, whichever is greater.

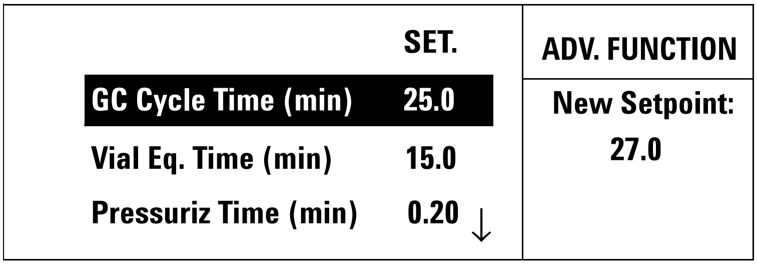
Enter a setpoint value of 0 to turn the zone off. The Headspace Sampler displays `OFF`.

As a general rule, the temperatures for the loop and transfer line should be set higher than the oven temperature to avoid condensation. Set the loop temperature 10 to 15 °C higher than the oven, and set the transfer line 20 to 25 °C higher than the oven.

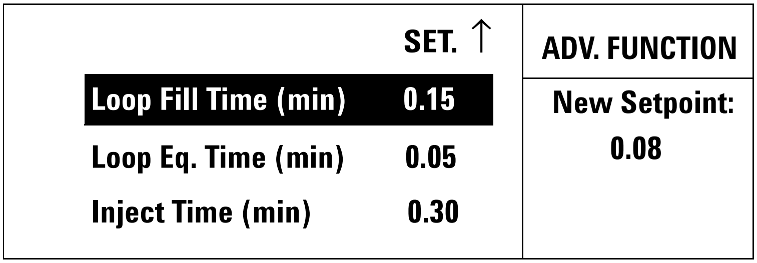
CAUTION

Oven temperature should not exceed 10 °C below the boiling point of any solvents in the vial. Exceeding this may cause the cap and septum to leak or to pop off ; the vial pressure line could also become contaminated.

Event Times



	SET.	ADV. FUNCTION
GC Cycle Time (min)	25.0	New Setpoint: 27.0
Vial Eq. Time (min)	15.0	
Pressuriz Time (min)	0.20 ↓	



	SET. ↑	ADV. FUNCTION
Loop Fill Time (min)	0.15	New Setpoint: 0.08
Loop Eq. Time (min)	0.05	
Inject Time (min)	0.30	

Description

Select `Event Times` from the Active Method menu to specify the duration of various method parameters.

GC Cycle Time The time required for the entire GC run to be completed. It is the sum of the GC run time and GC equilibration time (read from the GC front panel) and an estimate of the cool–down time required (if any). The GC cycle time must be long enough for the GC to become “ready”. This time allows the Headspace Sampler to introduce samples into the oven efficiently and avoid injecting a sample before the GC is ready.

Vial Eq. Time (vial equilibration time) Time the vial spends in the oven heating the sample. The proper length of time depends on the type of sample (solid, liquid), the amount of sample, and the partition coefficient of the analytes. In most cases, the vial equilibration time should be long enough to reach equilibrium in the vial.

Pressuriz. Time (pressurization time)

Time during which the vial pressurization gas is introduced into the sample vial to build up additional pressure in the vial.

Loop Fill Time Time in which the headspace/vial pressurization gas mixture passes through the sample loop to vent. This fills the sample loop with a precise amount of headspace gas.

Loop Eq. Time (loop equilibration time) Time after the vent valve closes to allow the analytes in the sample loop to equilibrate to the higher loop temperature and to allow pressure and flow in the loop to stabilize.

Inject Time Time in which the gases in the sample loop are injected into the GC. This time must be sufficient for complete sample transfer. If the time is too short, the analysis will lose sensitivity because not all the sample will be transferred. It is not a problem if the time is longer than necessary. The actual time needed depends on the carrier flow rate and the sample loop size.

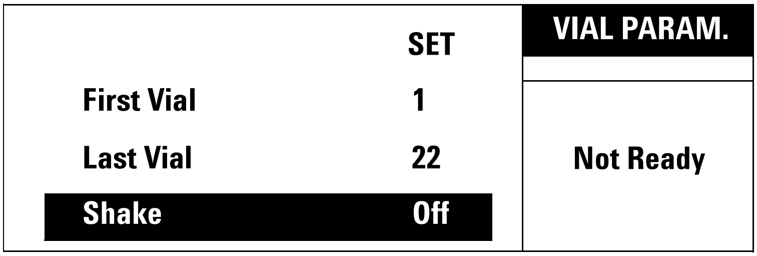
Procedure

Use the cursor keys to scroll through the list of event times. Enter a new time value with the numeric keypad. Press **Enter** to set the value. See Table 4 for setpoint ranges. See page 54 for descriptions of each event.

Table 4 Event time setpoints

Event	Allowed setpoint range (min. increments)	Default Value (min.)
GC Cycle Time	1.0–999.9 in 0.1	25
Vial Eq. Time	0–999.9 in 0.1	15
Pressuriz. Time	0–99.99 in 0.01	0.20
Loop Fill Time	0–99.99 in 0.01	0.20
Loop Eq. Time	0–99.99 in 0.01	0.05
Inject Time	0–99.99 in 0.01	1.00

Vial Parameters



VIAL PARAM.

	SET
First Vial	1
Last Vial	22
Shake	Off

Not Ready

Description

Select `Vial Parameters` from the Active Method menu to specify the vial(s) analyzed during a method and access the shake function.

First Vial Designates the first vial (1–70) analyzed by the current method.

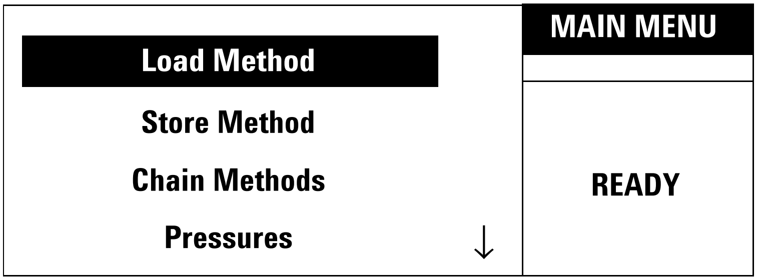
Last Vial Designates the last vial (1–70) analyzed by the current method. The last vial number cannot be lower than the first vial number. The Headspace Sampler will give an error message and you will need to re-enter the vial number.

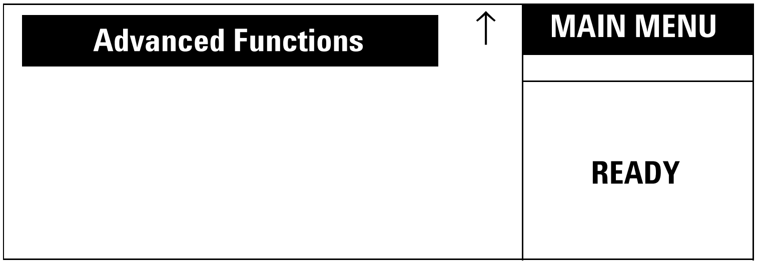
Shake Shake specifies the agitation level of the oven carousel during vial equilibration. Shaking the vial may shorten the needed vial equilibration time.

There are three shake settings: off, low, and high. Specify the level desired using the cursor keys followed by **Enter**. Low is sufficient for most applications.

Menu Key

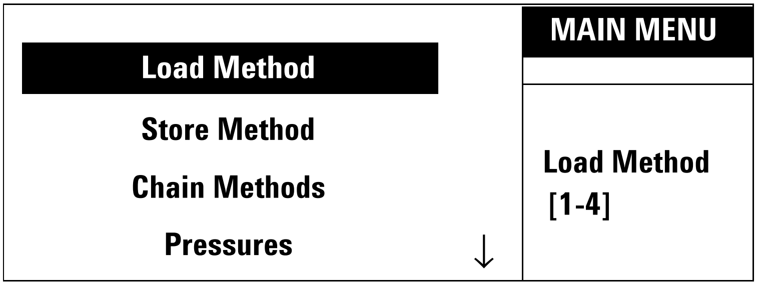
Press the **Menu** key to display the following screen:


Load Method
Store Method
Chain Methods
Pressures
↓
MAIN MENU
READY


Advanced Functions
↑
MAIN MENU
READY

- For more information on Load Method, see page 59.
- For more information on Store Method, see page 60.
- For more information on Chain Method, see page 61.
- For more information on Pressures, see page 62.
- For more information on Advanced Functions, see page 64.

Load Method


MAIN MENU
Load Method
Store Method
Chain Methods
Pressures
↓
Load Method
[1-4]

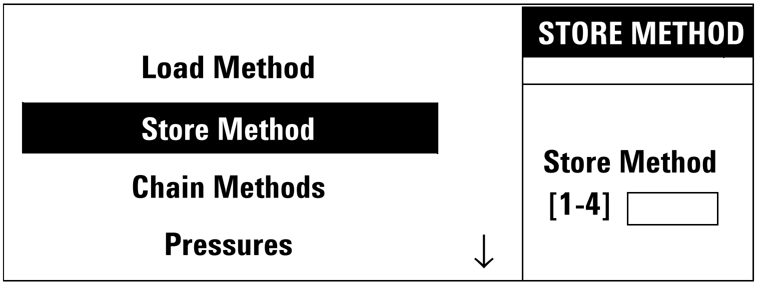
Description

Load Method retrieves one of four stored methods and places it into active memory for use in the current analysis. Note that loading a method overwrites the current method in active memory.

Procedure

Use the numeric keypad to enter a number between 1 and 4. Press the **Enter** key to load the method stored in that location.

Store Method


Load Method
Store Method
Chain Methods
Pressures
↓
STORE METHOD
Store Method
[1-4]

Description

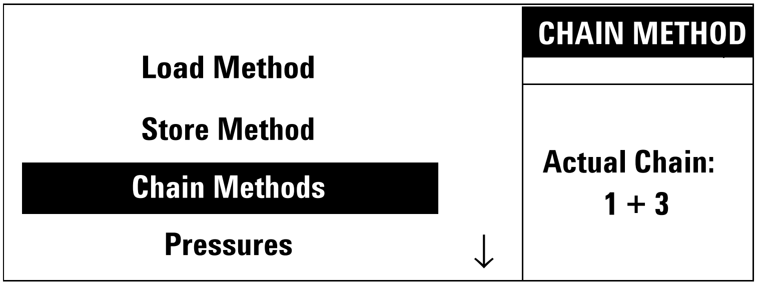
Store Method stores the current method in one of four method storage areas.

Procedure

Use the numeric keypad to enter a number between 1 and 4. Press the **Enter** key to store the method in that storage area.

CAUTION Storing a method overwrites any method already stored in that storage area. There is no warning message that an overwrite is taking place.

Chain Methods


CHAIN METHOD
Load Method
Store Method
Chain Methods
Pressures
↓

Actual Chain:
1 + 3

Description

Chain Methods uses two to four methods sequentially to analyze groups of samples in the tray. Chain Methods is disabled if Parameter Increment is enabled. Note that chaining methods overwrites the current method in active memory.

Procedure

Use the numeric keypad to enter two to four numbers between 1 and 4. Press **Enter** to chain the methods together.

Enter **0** on the numeric keypad to disable the chain.

Any combination of methods is allowed. Some examples include:

1+1+1+1

1+2+3+4

4+2+3+2

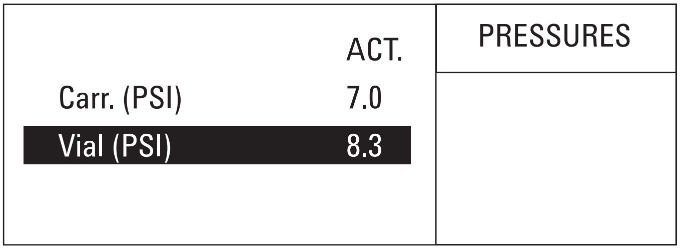
2+1

3+1+2

Pressures

This screen displays the pressure of the carrier gas or the vial pressurization gas. The carrier gas pressure is measured at the outlet of the flow controller. The vial pressurization is measured at the outlet of the pressure regulator before the in-line restrictor. See page 32 for a diagram of the pneumatics system and the location of the pressure sensor boards.

The pressures screen is a read-only display. Use the cursor keys to scroll the display from the carrier gas to the vial pressurization gas. The maximum transducer pressure is 100 psig.



PRESSURES

	ACT.
Carr. (PSI)	7.0
Vial (PSI)	8.3

4

Advanced Functions

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List of Advanced Functions 65
Advanced Function Descriptions 67
  Stabilize Time 67
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  Multiple Headspace Extraction 75
  Check for Ready 76
  Reset 78
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  Zone Calibration 81
  LAN Configuration 82
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  Vent Valve purging 84
  Leak test 86
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This chapter describes the nonroutine functions controlled by the Advanced Function menu. It describes the displays, value ranges, and how to edit the setpoints for these functions.

Using an Advanced Function

To access an advanced function setpoint or reading:

1. Press the **Menu** key.
2. Scroll to Menu item `Advanced Functions` and press **Enter**.
3. Scroll to the desired function and press **Enter** or press its corresponding menu number on the keypad. If the menu number is greater than 9, use a decimal point followed by the second digit of the menu number. For example, press **. 2** to select menu item 12.
4. Some Advanced functions contain subfunctions, such as Zone Calibration. Scroll to the desired subfunction and press **Enter**, if applicable.

At any point in a menu, press **Clear** to return to a higher-level display.

See Table 5 for a quick reference of the Advanced functions. See "Advanced Function Descriptions" on page 67 for details.

List of Advanced Functions

Table 5 Quick reference of Advanced functions

Display	Definition/Function	Menu selection keys	Page
STABILIZE TIME	Sets wait time between oven reaching initial setpoint and first vial loading	**1**	67
PARAM. INCREMENT	Creates stepwise increments in oven temperature or equilibration time for subsequent vials	**2**	68
VIAL NO. AND LOC.	Displays the vial number in each oven carousel location. Vial numbers correspond to the original tray position of the vial.	**3**	70
KEYBOARD LOCK	Disables all keyboard functions except navigation and method start/stop	**4**	71
MANUAL OPERATION	Allows the user to manually move all motors, lifters, the tray, and the shutter, and open and close all valves on the Headspace Sampler	**5**	72
PRESSURE UNITS	Changes the pressure units	**6**	73
STORED METHOD	Load one of five preset methods	**7**	74
MULTI. HS EXTR.	Sets parameters for multiple headspace extraction	**8**	75
CHECK FOR READY	Tells the HS whether or not to look for a ready signal from the GC before injecting	**9**	76
RESET	Select Reset to force the sampler to abort whatever it is doing and rerun its startup routine. Vials return to the tray.	**., 0**	78
VALVE COUNT	Use this function to view/reset the number of valve cycles for the sample inject valve	**., 1**	79
VIAL SIZE	Choose 10-mL or 20-mL vials	**., 2**	80
ZONE CALIBRATION	Create a custom temperature offset for the oven and sample loop heated zones	**., 3**	81
LAN CONFIGURATION	Sets the IP Address, subnet mask, and gateway	**., 4**	82
MAC ADDRESS	Displays the MAC Address	**., 5**	83

Table 5 Quick reference of Advanced functions (continued)

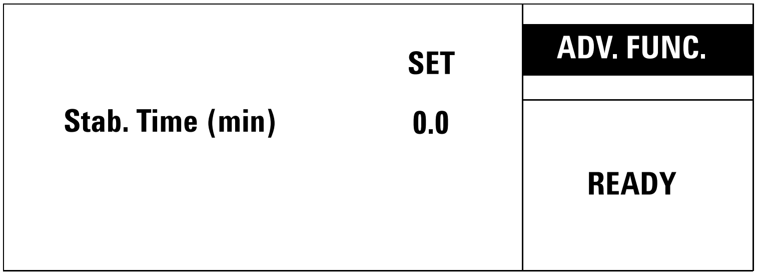
ENABLE RS-232	Sets the Headspace Sampler to RS-232 or LAN mode. This function requires a power cycle when switching from RS232 to LAN.	., **6**	83
PURGE VALVES	Forces pressurization gas out the vent to purge the line	., **7**	84
LEAK TEST	Starts running the leak test	., **8**	86
DIAGNOSTIC	Displays firmware revision, RAM/ROM status, and serial number	., **9**	87

Advanced Function Descriptions

Stabilize Time

Description

This function changes the length of time the oven equilibrates after reaching its initial setpoint before vials are loaded into the oven. Selecting Stabilize Time displays the following:



SET

Stab. Time (min) 0.0

ADV. FUNC.

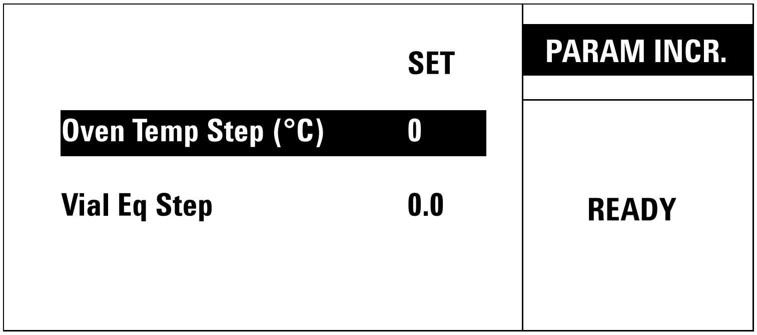
READY

Procedure

Enter the number on the keypad and then press **Enter** to set the value.

One minute is the default time. The allowed range of values is 0.0 to 99.9 minutes.

Parameter Increment



	SET
Oven Temp Step (°C)	0
Vial Eq Step	0.0

PARAM INCR.

READY

Description

Parameter Increment is a method development tool that automatically makes stepwise increases in either vial equilibration time or oven temperature for each subsequent vial sampled.

During parameter incrementing, each vial is sampled and returned to the carousel. The sampler displays NOT READY for the duration of the GC Cycle Time between each increment. If Vial Temp is incremented, the display reads WAITING FOR READY until the new setpoint is reached.

The default values for both displays are 0.0, which means that the parameter increment is off. The two parameters are mutually exclusive: if a nonzero number is entered into one, the other is automatically set to zero.

Oven Temp Step Entering a value for the Oven Temp Step increases the Vial Temperature during vial equilibration by that many degrees for the second vial and each subsequent vial in the present method.

For example, enter an oven temperature step of 4 °C and run a method with an oven temperature of 100 °C. The first vial has an oven temperature of 100 °C, the second vial has an oven temperature of 104 °C, and the third vial has an oven temperature of 108 °C.

The value range is from 0 to 99 °C.

Vial Eq Step Entering a value for the Vial Eq Step increment increases the stabilization time by that many minutes for the second vial and each subsequent vial in the present method.

The value range is 0.0 to 99.9 minutes.

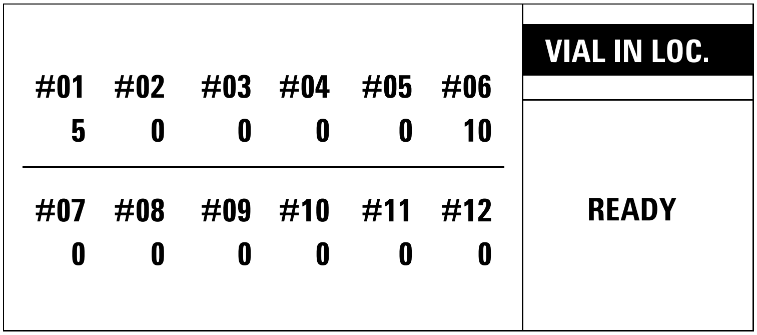
Procedure

When enabled, use the cursor keys to scroll to the oven temperature and stabilize time step increments:

Change the increments from the numeric keypad and then press the **Enter** key to set the change in value.

- Do not allow the maximum vial temperature to be closer than 10 °C to the boiling point of any solvent in the sample.
- The Vial Temp Incr function increases the temperature from run to run in the headspace vial only. The loop and transfer line temperatures remain at their original setpoints. Be sure to set the loop and transfer line temperatures so they exceed the highest temperature the vial is programmed to reach. Take note of any maximum temperature on the transfer line you are using.
- Chain methods are not allowed if either the Vial Temp Incr or the Vial Equilibration Incr is set to a non-zero value.
- If the combination of oven temperature step and number of vials sets the oven temperature above the maximum temperature allowed (for example, the open temperature is 155 °C, the step is 10 °C, and there are ten vials), the HS displays a warning message. If you start the run anyway, the analysis stops when the oven reaches 230 °C

Vial number and location



VIAL IN LOC.

#01	#02	#03	#04	#05	#06
5	0	0	0	0	10

#07	#08	#09	#10	#11	#12
0	0	0	0	0	0

READY

Description

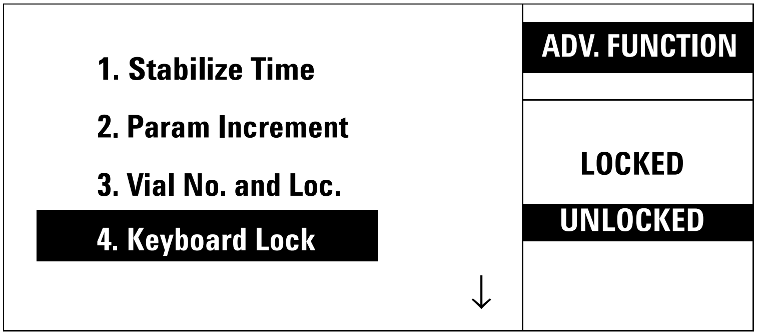
Vial number and location tells the user what vial number is in which position in the oven carousel. Vial numbers correspond directly to the original tray position of the vial. For example, vial 1 is the vial placed into tray position 1.

The first number on the left of the screen is the first oven carousel position. The last number on the right of the screen is the sixth oven carousel position. In this example, vial number five is in oven carousel position one and vial number 10 is in position six. This is a read-only display. No editing is possible.

Keyboard Lock

Description

This function disables input from the numeric keypad and Enter keys. They keys can still be used for navigation and starting a method. Selecting Keyboard Lock displays a small graphic of a lock on the top level screen.

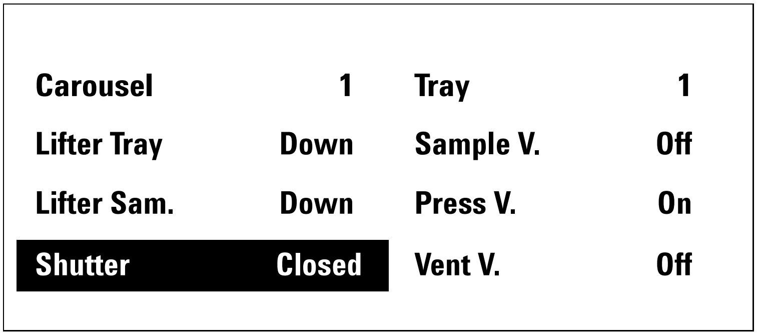

1. Stabilize Time
2. Param Increment
3. Vial No. and Loc.
4. Keyboard Lock
↓
ADV. FUNCTION
LOCKED
UNLOCKED

Procedure

To lock the keyboard, use the cursor keys to scroll to LOCKED and press **Enter**.

Scroll to UNLOCKED and press **Enter** to unlock the keyboard.

Manual Operation



Carousel	1	Tray	1
Lifter Tray	Down	Sample V.	Off
Lifter Sam.	Down	Press V.	On
Shutter	Closed	Vent V.	Off

Description

This function manually operates the vial carousel advance, the vial tray, valves, shutter, and both vial lifts to check for malfunctions. If the operations do not work, see Troubleshooting and Error Messages on page 163, then call Agilent service.

The motors are not reset to their default states after using Manual Operation. Use the reset function to restore motors to their default state. See page 78.

Procedure

Use the cursor keys to scroll through the menu of items.

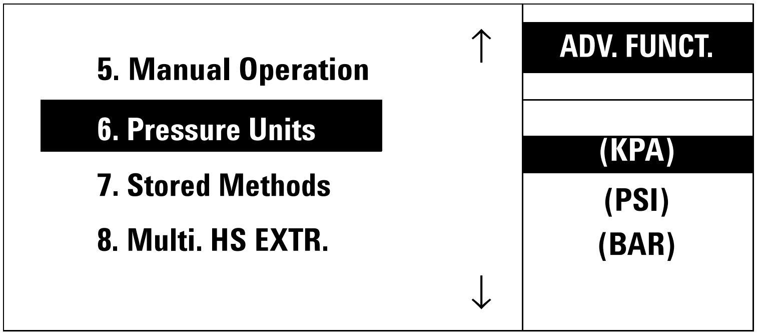
Carousel and Tray Press **1** to rotate clockwise 1 position. Press **0** to rotate counter-clockwise 1 position.

Lifter Tray and Lifter Sam. Press **1** to raise the lift. Press **0** to lower the lift. The display shows how far the lift is raised in relation to vial sizes. Note that the Lifter Tray cannot be raised while the shutter is closed.

Shutter Press **1** to open the Shutter to the mid position. The display shows `midway`. Press **1** a second time to open the Shutter. The display shows `open`. Press **0** to return the Shutter to the midway position, and press **0** a second time to close the Shutter. Note that the Shutter cannot be closed while the Tray Lifter is raised.

Sample V., Press V., and Vent V. Press **1** to turn the valve on. Press **0** to turn the valve off.

Pressure Units


5. Manual Operation
6. Pressure Units
7. Stored Methods
8. Multi. HS EXTR.
↑
↓
ADV. FUNCT.
(KPA)
(PSI)
(BAR)

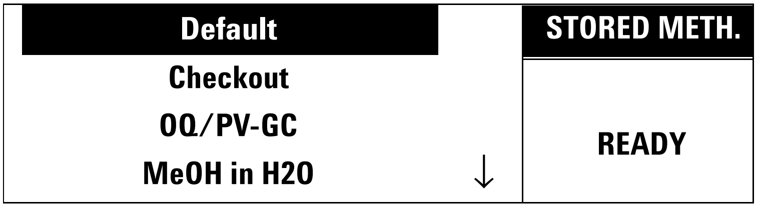
Description

This function changes the pressure units for the displays among psi, kPa, and bar.

Procedure

Psi is the default pressure unit when the HS powers up. Use the cursor keys to scroll to kPa or bar. When units are shown on the display, they are active.

Stored Method



Default
Checkout
OQ/PV-GC
MeOH in H2O ↓

STORED METH.

READY

Description

Use the Stored Method screen to load one of five preset methods. See Table 6 for the parameters stored in each method.

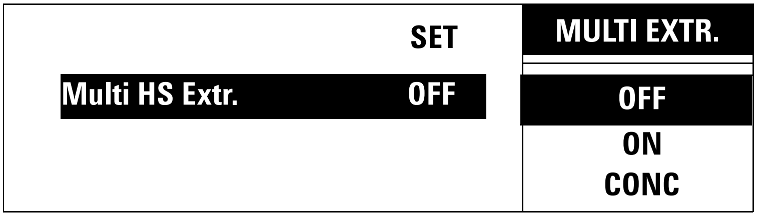
Table 6 Stored Methods

	Parameter	Default	Checkout	OQ/PV-GC	MeOH in H$_2$0	OQ/PV-MSD
Heated zones (°C)	Oven	50	100	100	60	100
	Loop	60	110	110	110	150
	Tr. Line	70	115	115	120	180
Equilibration time (min)	GC cycle time	25.0	5.5	2.0	2.0	6.0
	Vial eq. time	15.0	7.0	7.0	1.5	5.0
	Pressuriz. time	0.20	0.08	0.08	0.20	0.1
	Loop fill time	0.20	0.5	0.5	0.20	0.5
	Loop eq. time	0.05	0.05	0.05	0.05	0.1
	Inject time	1.0	1.0	0.2	0.25	1.0
Vial parameters	First vial	1	1	1	1	1
	Last vial	10	1	8	1	9
	Shake	off	off	off	off	off
	Vial size	20 mL	20 mL	20 mL	20 mL	20 mL
Advanced functions	Multiple extraction	off	off	off	on	off
	Injections per vial	1	1	1	20	1

Procedure

Use the cursor keys to scroll select one of five stored default methods. Press the **Enter** key to load the method.

Multiple Headspace Extraction



Multi HS Extr. OFF

Description

Multiple Headspace Extraction (MHE) is used to sample the same vial repeatedly. The vial is raised on to the sampling probe, sampled, and lowered off of the probe for each extraction. There are two types of MHE available on the Headspace Sampler.

- If MHE is set to “ON”, the HS sends a start signal to the GC after each extraction.
- If MHE is set to “CONC”, the HS sends a start signal to the GC after the final extraction.

NOTE MHE is not allowed if Parameter Increment is on. See page 68.

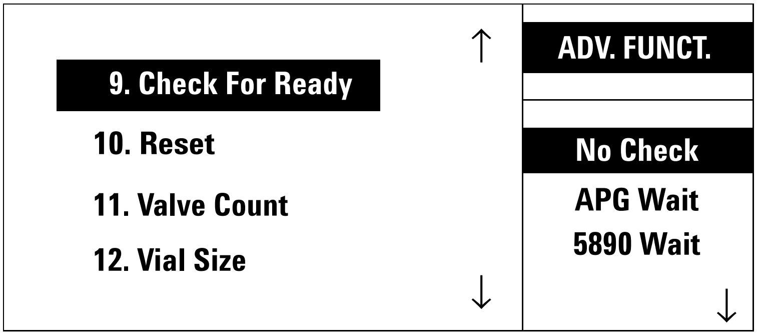
Procedure

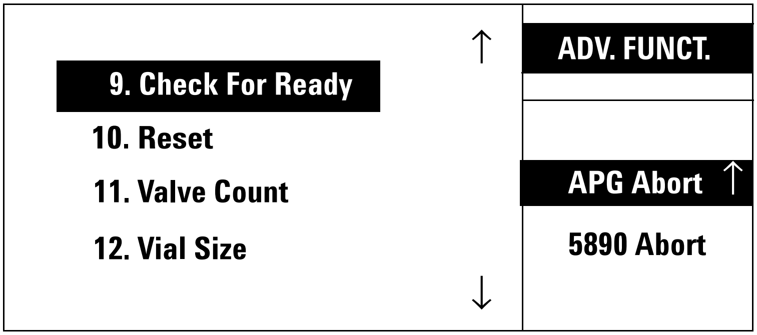
Use the cursor keys to select a menu item on the right.

OFF Press **Enter** to turn off MHE.

ON and **CONC** Press **Enter** to turn on MHE. Use the keypad to enter the number of extractions per vial. The allowed range is 2 -100 extractions.

Check for Ready


9. Check For Ready
10. Reset
11. Valve Count
12. Vial Size
ADV. FUNCT.
No Check
APG Wait
5890 Wait


9. Check For Ready
10. Reset
11. Valve Count
12. Vial Size
ADV. FUNCT.
APG Abort
5890 Abort

Description

A hardwired connection is built into the remote start cable between the GC and the Headspace Sampler. Use this function to wait for the GC ready signal, abort the sequence, or proceed without the GC ready signal.

If Check For Ready is active, then the sampler will check for the presence of a ready signal from the GC just prior to injection. If a GC ready signal is not found, the sampler waits, aborts, or continues depending on the mode.

The following modes are available:

No Check The HS injects regardless of the ready state of the GC.

5890 Wait The HS waits for 5890 GC ready signal before beginning the sequence. The Headspace Sampler's Sample Equilibration Time is ignored; the sequence is controlled by GC readiness. This mode corrupts constant heating time if the GC cycle time is not complete when the constant heating time is reached.

APG Wait The HS waits for 6850/6890/6820 GC ready signal before beginning the sequence. The Headspace Sampler's Sample Equilibration Time is ignored; the sequence is controlled by GC readiness. This mode corrupts constant heating time if the GC cycle time is not complete when the constant heating time is reached.

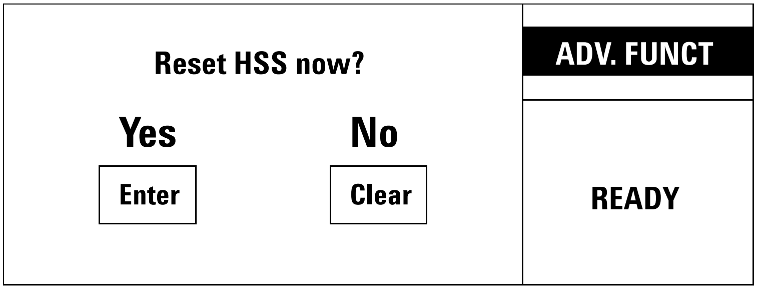
5890 Abort The sequence aborts if the 5890 GC is not ready when the injection is about to occur. All vials return to the tray, and the HS displays the message GC NOT READY. This is the recommended setting for users with a 5890 GC.

APG Abort The sequence aborts if the 6850/6890/6820 GC is not ready when the injection is about to occur. All vials return to the tray, and the HS displays the message GC NOT READY. This is the recommended setting for users with a 6850, 6890, or 6820 GC.

Procedure

Use the cursor keys to highlight the desired mode in the right section of the display screen. Press **Enter** to select the highlighted mode.

Reset


Reset HSS now?

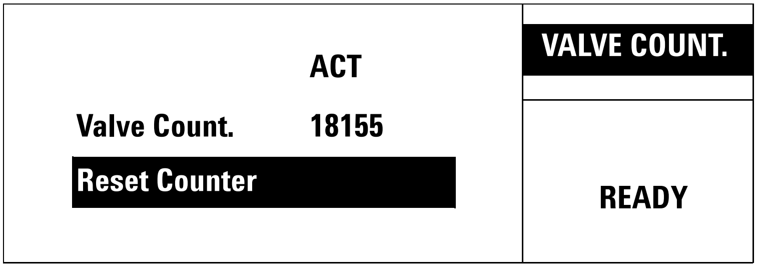
Description

Select Reset to force the sampler to abort whatever it is doing and rerun its startup routine. See “Power on diagnostics” on page 35 for more information.

Note:

- The instrument remains Not Ready until all setpoints for the method are reached.
- The last method in use before the reset remains in effect.
- After the reset, the Headspace Sampler asks if it should recover vials from the oven carousel. Choose “yes” to have the Headspace Sampler return the vials to the tray. Choose “no” to manually recover the vials if any remain in the oven.

Valve Count



ACT

Valve Count. 18155

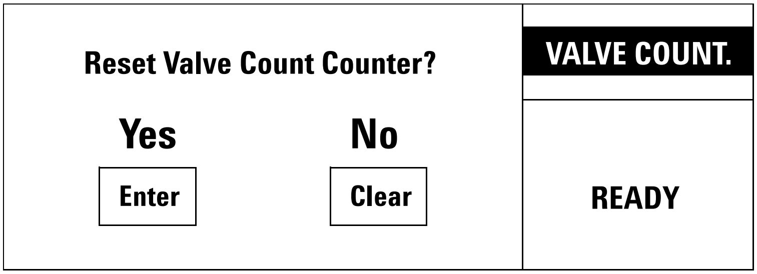
Reset Counter
VALVE COUNT.
READY

Description

Valve Count displays the number of times the sampling valve has actuated. Agilent service personnel may refer to the Valve Count to get an idea of the extent of the sampler's use. A valve must move in both directions (on and off) before the counter increments.

Procedure

To reset the counter, use the cursor keys to highlight Reset Counter. Press **Enter**. The following screen appears:

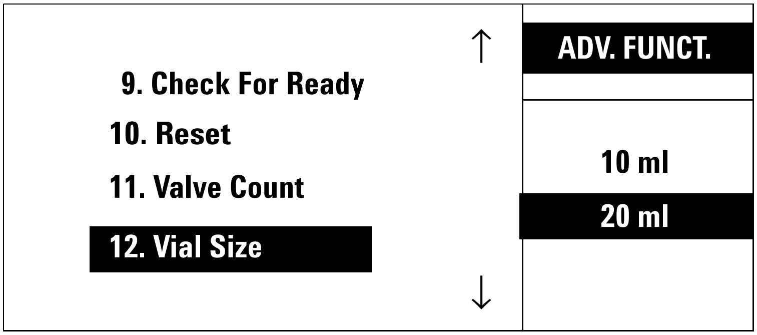


Reset Valve Count Counter?

Yes
No
Enter
Clear
VALVE COUNT.
READY

Press **Enter** to reset the counter or **Clear** to return to the previous screen.

Vial Size


9. Check For Ready
10. Reset
11. Valve Count
12. Vial Size
ADV. FUNCT.
10 ml
20 ml

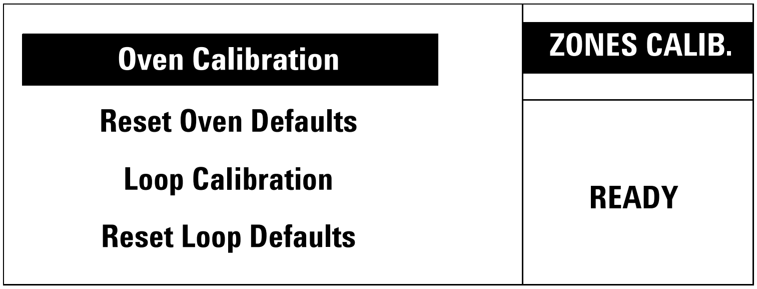
Description

This function sets the HS for use with 10-mL or 20-mL vials. The HS cannot use 10-mL and 20-mL vials together in the same method.

Procedure

Use the cursor keys to select the appropriate Vial Size and press **Enter**.

Zone Calibration


Oven Calibration
Reset Oven Defaults
Loop Calibration
Reset Loop Defaults
ZONES CALIB.
READY

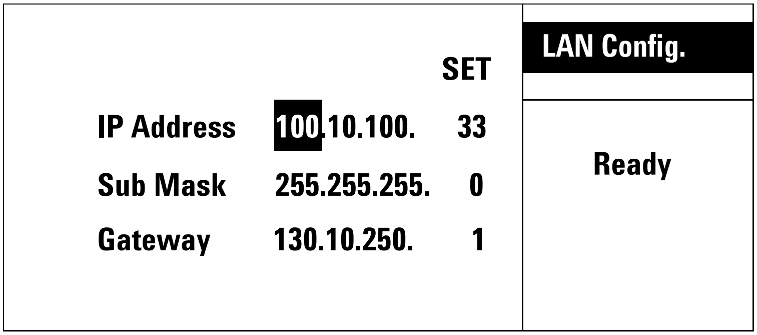
Description

The Zones Calibration screen creates a custom temperature offset for the Headspace Sampler. The calibration can also be restored to factory defaults at this screen.

The menu choices Reset Oven Defaults and Reset Loop Defaults only appear if a custom offset exists.

See Zone Calibration on page 156 for more of the calibration procedure.

LAN Configuration



LAN Config.

SET

IP Address 100.10.100. 33

Sub Mask 255.255.255. 0

Gateway 130.10.250. 1

Ready

Description

This function is used to configure the Headspace Sampler for use on a LAN.

Procedure

Use the cursor keys to scroll through numbers in the IP Address, Sub Mask, and Gateway.

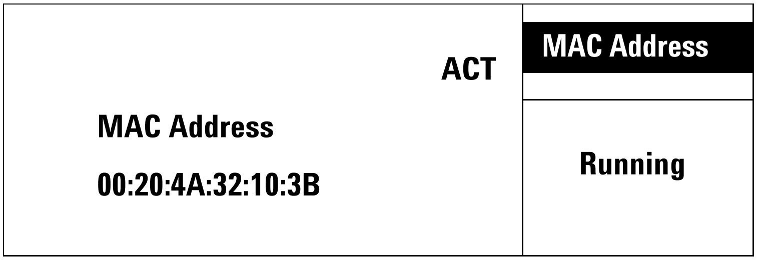
Key in a number on the numeric keypad to change the highlighted value on the screen. Press **Enter** to accept the number or **Clear** to cancel.

The highlight automatically moves to the next value after entering a number.

Enable RS232 must be turned off before using the Headspace Sampler on a LAN. See Enable RS232 on page 83 for details.

The Headspace Sampler must be reset to activate the LAN connection when switching from RS232 to LAN.

MAC Address



ACT

MAC Address

00:20:4A:32:10:3B

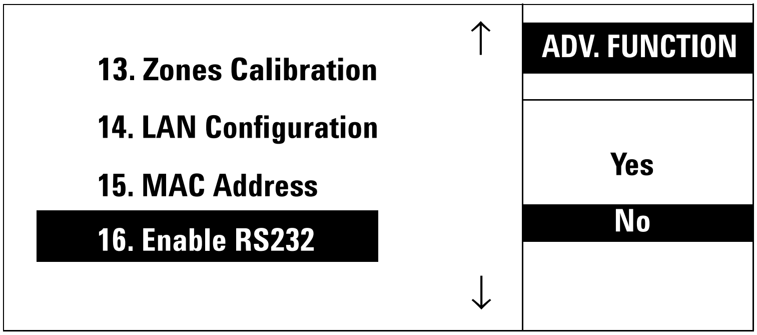
MAC Address

Running

Description

MAC Address is a read only display.

Enable RS232



13. Zones Calibration
14. LAN Configuration
15. MAC Address
16. Enable RS232

ADV. FUNCTION

Yes

No

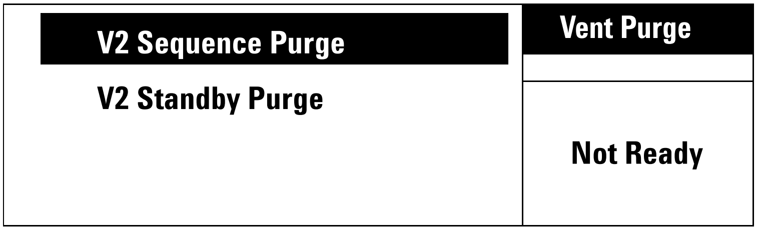
Description

This function enables the RS232 port on the Headspace Sampler. Disable the RS232 port to enable the LAN port.

Procedure

Use the cursor keys to select “Yes” to enable RS232, or “No” to disable RS232 and enable the LAN port.

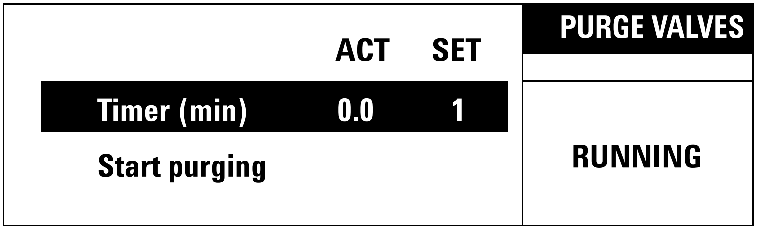
Vent Valve purging


V2 Sequence Purge

V2 Standby Purge

Vent Purge

Not Ready


	ACT	SET
Timer (min)	0.0	1
Start purging		

PURGE VALVES

RUNNING

Description

V2 Sequence Purge This function sets the amount of time that the V2 vent valve is open after each vial injection. The default value is 30 second.

V2 Standby Purge This function opens the vent valve and allows pressurization gas to escape through the vent. Use this function to remove any traces of unwanted material from the Headspace Sampler plumbing before running a method, if desired.

Procedure

V2 Sequence Purge Select `V2 Sequence Purge` with the cursor keys and press **Enter**. Use the key pad to key in a time value. Press **Enter**.

V2 Standby Purge Select `V2 Standby Purge` with the cursor keys and press **Enter**. Use the key pad to key in a time value between 1 and 999 minutes. Press **Enter**.

The value appears in the SET column. The ACT column begins counting down the remaining minutes in real time. The process ends automatically when the timer reaches 0.

Leak test

Description

A leak test checks for the following:

- Restrictions in the flow path associated with filling the loop
- Leaks in the carrier flow path
- Leaks in the vial pressurization flow path
- Leaks across the 6-port sampling valve

Symptoms Symptoms associated with restrictions include:

- No peaks
- Loss of sensitivity

Restrictions are often caused by the swelling of the solenoid vent valve seals or the build up of residues in the vent tube.

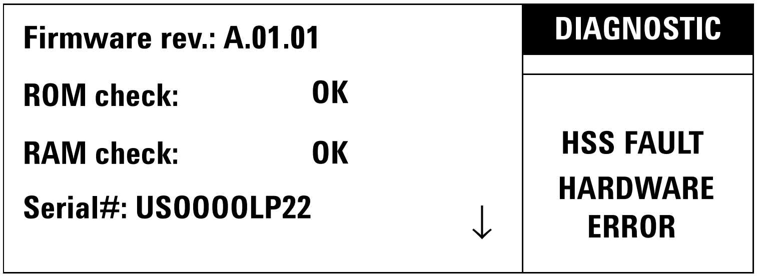
Symptoms associated with leaks include:

- High detector backgrounds
- Loss of sensitivity

The most common leaks are at the connections of the transfer line, external plumbing, and sample probe union.

See Leak Test on page 148 for the leak test procedure.

Diagnostic



Firmware rev.: A.01.01

ROM check: OK

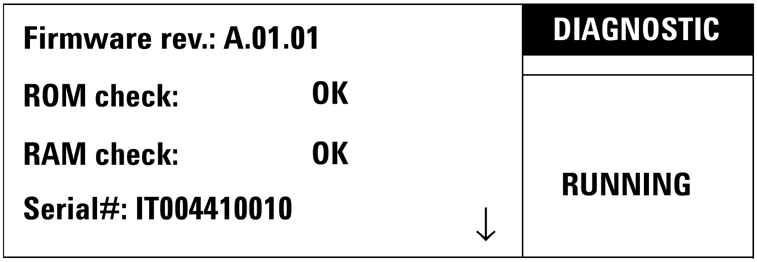
RAM check: OK

Serial#: USOOOOLP22 ↓

DIAGNOSTIC

HSS FAULT

HARDWARE
ERROR



Firmware rev.: A.01.01

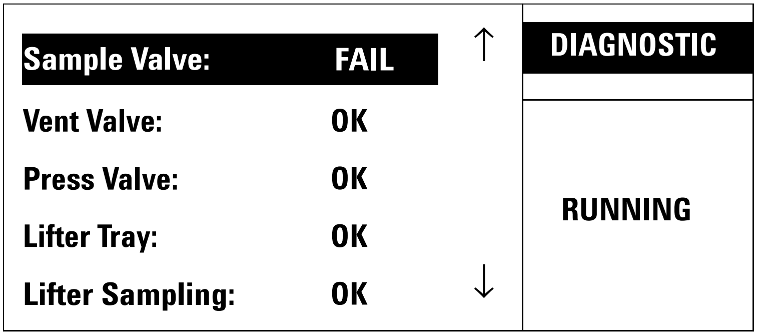
ROM check: OK

RAM check: OK

Serial#: IT004410010 ↓

DIAGNOSTIC

RUNNING



↑

Sample Valve: FAIL

Vent Valve: OK

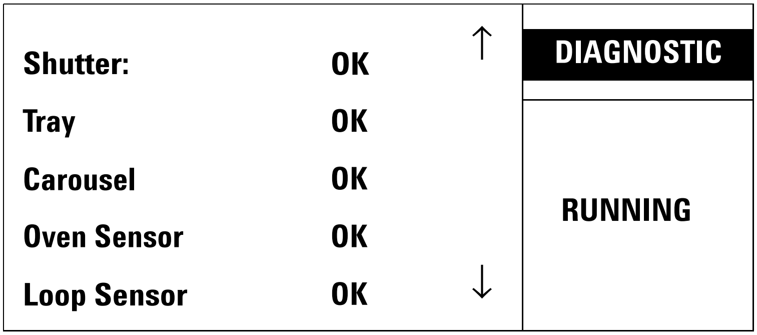
Press Valve: OK

Lifter Tray: OK

Lifter Sampling: OK ↓

DIAGNOSTIC

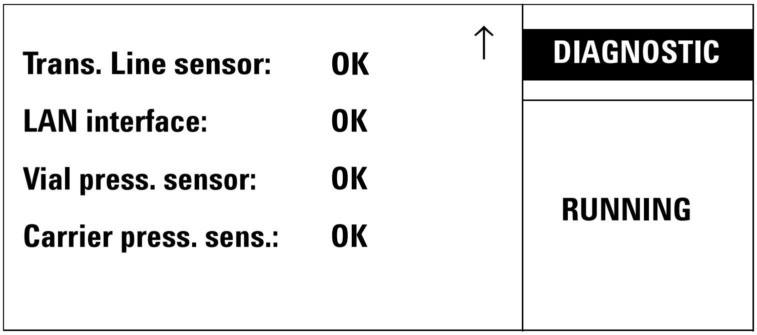
RUNNING



Shutter: OK ↑
Tray OK
Carousel OK
Oven Sensor OK
Loop Sensor OK ↓

DIAGNOSTIC

RUNNING



Trans. Line sensor: OK ↑
LAN interface: OK
Vial press. sensor: OK
Carrier press. sens.: OK

DIAGNOSTIC

RUNNING

Description

The diagnostic screen is a read only display that shows the power-on system diagnostic messages.

5

Preparing a Sample

Sample Preparation 90
Loading the Sample Tray 92

This chapter describes the steps necessary to prepare a sample for analysis, including filling and sealing a sample vial and loading the sample tray.

Sample Preparation

Place samples for headspace extraction in glass vials. Place the glass vials in the Headspace Sampler tray. In general, fill sample vials about half way (although vials can be filled to the 3/4 level). This ensures that the sampling probe will not come into contact with the solid or liquid phase during the sampling step.

The G1888 Network Headspace Sampler accepts either 10-mL or 20-mL sample vials. The Headspace Sampler can only run one vial size at a time. See Vial Size on page 80 for details on changing the vial size.

The vial must be sealed properly to insure that the headspace gases do not escape prematurely. For crimp top vials, use a crimper (Agilent part no. 9301–0720) designed for headspace vials with 20-mm caps to seal the vials. Screw caps (Agilent part no. 5188–2759) and screw top vials (Agilent part no. 5188–2753) are also available . See the list of consumables on page 127.

NOTE Sampling from an incorrectly capped vial may cause the Headspace Sampler to jam during septum puncture.

Do the following to prepare a vial for sampling:

1. Place a septum in a vial cap with the Teflon side facing the vial. Take care not to contaminate the septum.
2. Place the cap upside down on a table.
3. Place the sample in the vial. Most vial should not be more than 50% full, but some vials can reach 75% full.
4. Place the septum and cap together over the vial opening.

5 Place the crimper over the cap and vial. See Figure 14.

6 With slow and steady pressure, squeeze the crimper handles to seal the vial.

7 Remove the crimper.

8 Turn the vial and cap 90° and crimp a second time.

9 Check that the seal is adequate by trying to twist the cap on the vial. It should be difficult or impossible to turn the cap. If the cap turns easily, recrimp the cap. The crimping tool may need adjustment.

10 To decap vials, use a decapper for vials with 20-mm caps (part no. 5181–1214).

NOTE

Vials will contain laboratory air, which may be contaminated with things such as solvents or freon leaking from air conditioners. If contaminants will adversely affect your results, use argon to purge the vials just before capping and sealing.

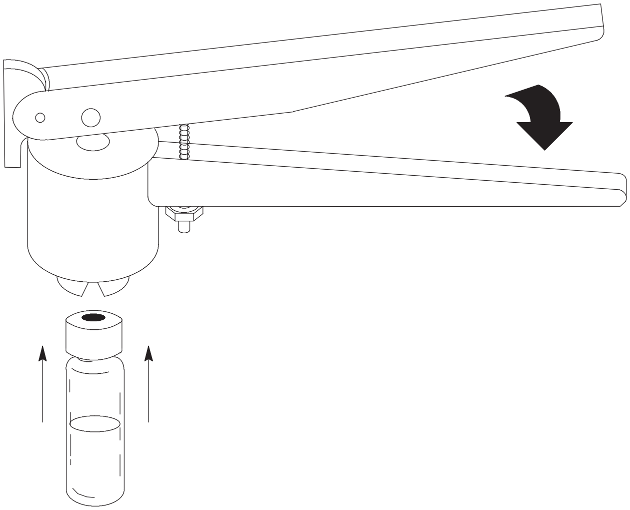


Figure 14 Position of crimper and vials for proper capping

Loading the Sample Tray

Press **Advance Tray**, {position+7}, and **Enter** to move the desired loading position to the very front of the sample tray.

Using the cursor keys, advance tray positions one-by-one while adding vials until all samples are loaded into the tray.

Do not mix 10-mL and 20-mL vials in a tray.

6

Entering a Method

Entering a Method 94
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  Entering Zone Temperatures 95
  Setting Event Times 97
  Setting Vial Parameters 100
Storing and Loading Methods 102
  Storing a method 102
  Loading a method 103
Chaining Methods 104
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This chapter details the procedure for entering a method, including storing and retrieving a method, and chaining methods.

Entering a Method

How to create a method

To create a method for the Headspace Sampler via the keypad, enter values for each setpoint in the following menu screens:

- Zone Temps: see page 95
- Event Times: see page 97
- Vial Parameters: see page 100

Each of these screens contains a number of setpoints. To enter a value for a setpoint, use the cursor keys to scroll to that setpoint, key in the value using the numeric keypad, then press **Enter**. If the value is not within the allowed range, the sampler displays a message showing the allowed range. Press any non-numeric key to remove the error message.

Press **Clear** to remove an incorrectly typed keystroke while entering setpoint values. The **Clear** key also returns to the top level of the display when a setpoint is not being entered.

Users may also set the vent valve purge time. Increasing the vent purge time may reduce carryover and contamination. See Vent Valve purging on page 84 for details.

After each setpoint is defined, store the method in one of four storage locations. See page 102 for more information on storing methods. Note that vial size is not stored as part of the method.

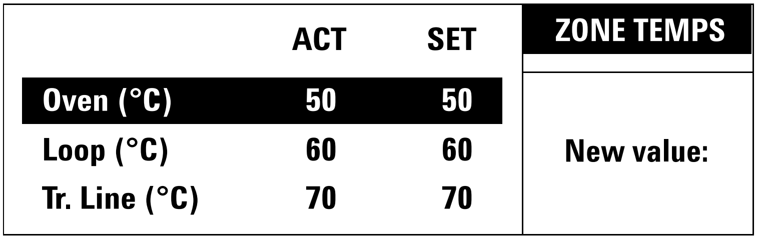
To run a sample, load the vials into the proper spots in the sample tray then press **Start**.

Entering Zone Temperatures

Use the ZONE TEMPS screen to enter values for the oven temperature, loop temperature, and transfer line temperature.

Navigate to the ZONE TEMPS screen

Press the **Active Method** key. Select `Zone Temps` from the Active Method menu. The sampler displays the following:



	ACT	SET
Oven (°C)	50	50
Loop (°C)	60	60
Tr. Line (°C)	70	70

ZONE TEMPS

New value:

For more information on zone temperatures, see Zone Temps on page 52.

Setting the oven zone temperature

Use the cursor keys to highlight `Oven (°C)`. Key in the desired value for the oven temperature setting, followed by **Enter**.

The allowed range for this setting is 40-230 °C in increments of 1 °C.

CAUTION

Oven temperature should not exceed 10 °C below the boiling point of any solvents in the vial. Exceeding this may cause the cap and septum to leak or to pop off ; the vial pressure line could also become contaminated.

NOTE

Although the sampler may allow you to enter temperatures from 0 °C to 39 °C, the minimum oven temperature is 40 °C or 15 °C above ambient, whichever is greater.

Setting the sample loop zone temperature

Use the cursor keys to highlight `Loop (°C)`. Key in the desired temperature, followed by **Enter**.

The allowed range for this setting is 45-250 °C in increments of 1 °C.

Setting the transfer line zone temperature

Use the cursor keys to highlight `Tr. Line (°C)`. Key in the desired temperature, followed by **Enter**.

The allowed range for this setting is 50-250 °C in increments of 1 °C.

NOTE

As a general rule, the loop and transfer line temperatures should be set higher than the oven temperature to avoid condensation. Set the loop temperature 15 °C higher than the oven, and set the transfer line 25 °C higher than the oven.

Setting Event Times

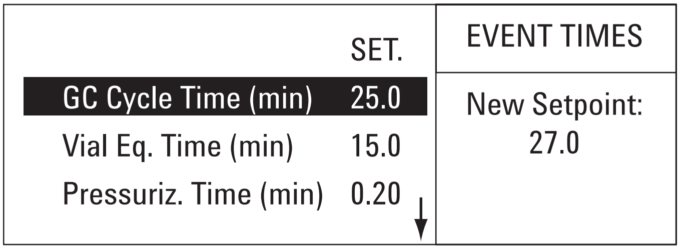
Use the EVENT TIMES screen to enter values in minutes for the timed events which make up the vial sampling sequence. The following events are included:

- GC Cycle Time
- Vial Equilibration Time
- Pressurization Time
- Loop Fill Time
- Loop Equilibration Time
- Inject Time

These times may be checked on the display and changed while the Headspace Sampler is running samples. Changes are applied to the next vial analyzed. You cannot change parameters for a vial currently being sampled. The keys involved are locked.

Navigate to the EVENT TIMES screen

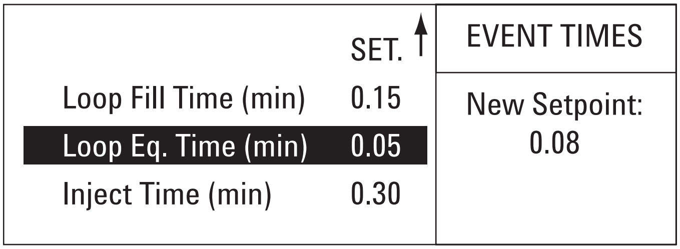
Press the **Active Method** key. Select `Event Times` from the Active Method menu. The sampler displays the following:



EVENT TIMES

	SET.
GC Cycle Time (min)	25.0
Vial Eq. Time (min)	15.0
Pressuriz. Time (min)	0.20

New Setpoint:
27.0



EVENT TIMES

	SET.
Loop Fill Time (min)	0.15
Loop Eq. Time (min)	0.05
Inject Time (min)	0.30

New Setpoint:
0.08

For more information on even times, see Event Times on page 54.

Setting the GC Cycle Time

The GC Cycle Time is the time required to complete the entire GC run. It is the sum of the GC run time, the GC equilibration time (read from the GC front panel), and an estimate of the cool-down time required (if any).

Use the cursor keys to highlight `GC Cycle Time (min)`. Key in the desired value, followed by **Enter**.

The allowed range for this setting is 1.0-999.9 minutes in increments of 0.1 minutes. The recommended minimum GC cycle time is 2.5 minutes.

Setting the Vial Equilibration Time

The Vial Equilibration Time is the time the vial spends in the oven heating to form the headspace gas.

Use the cursor keys to highlight `Vial Eq. Time (min)`. Key in the desired value, followed by **Enter**.

Vial equilibration time may be reduced for liquid samples if shake is enabled. See Vial Parameters.

The allowed range for this setting is 0-999.9 minutes in increments of 0.1 minutes.

Setting the Pressurization Time

Pressurization Time is the time the vial pressurization gas is introduced into the sample vial to build up additional pressure in the vial.

Use the cursor keys to highlight `Pressuriz. Time (min)`. Key in the desired value, followed by **Enter**.

The allowed range for this setting is 0-99.99 minutes in increments of 0.01 minutes.

Setting the Loop Fill Time

The Loop Fill Time is the time the headspace/vial pressurization gas mixture passes through the sample loop to vent. This fills the sample loop with a precise amount of headspace gas.

Use the cursor keys to highlight `Loop Fill Time (min)`. Key in the desired value, followed by **Enter**.

The allowed range for this setting is 0-99.99 minutes in increments of 0.01 minutes.

Setting the Loop Equilibration Time

The Loop Equilibration Time is the time after the vent valve closes. This allows the analytes in the sample loop to equilibrate to the higher loop temperature and allows the pressure and flow in the loop to stabilize.

Use the cursor keys to highlight `Loop Eq. Time (min)`. Key in the desired value, followed by **Enter**.

The allowed range for this setting is 0-99.99 minutes in increments of 0.01 minutes.

Setting the Inject Time

The Inject Time is the time the sample valve is open and the gases in the sample loop sweep into the GC.

Use the cursor keys to highlight `Inject Time (min)`. Key in the desired value, followed by **Enter**.

The allowed range for this setting is 0-99.99 minutes in increments of 0.01 minutes.

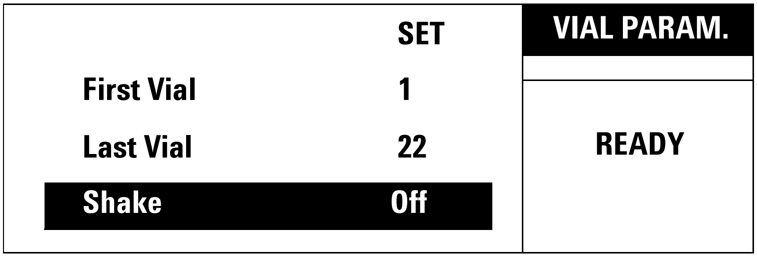
Setting Vial Parameters

Use the VIAL PARAM. screen to select sample tray locations used in the method and enable or disable vial shaking. The following setpoints are included:

- First vial
- Last vial
- Shake

Navigate to the VIAL PARAM. screen

Press the **Active Method** key. Select `Vial Param.` from the Active Method menu. The sampler displays the following:



	SET
First Vial	1
Last Vial	22
Shake	Off

VIAL PARAM.

READY

For more information on vial parameters, see Vial Parameters on page 57.

Setting the First Vial

First Vial specifies the location of the first vial to be analyzed by this method.

Use the cursor keys to highlight `First Vial`. Key in the desired location, followed by **Enter**.

The allowed range for this setting is 1-70.

Setting the Last Vial

Last Vial specifies the location of the last vial to be analyzed by this method.

Use the cursor keys to highlight `Last Vial`. Key in the desired location, followed by **Enter**.

The allowed range for this setting is 1-70. The number of the Last Vial cannot be lower than the number of the First Vial. If the Last Vial location is lower than the First Vial location, the Headspace Sampler gives an error message and the Last Vial location must be re-entered.

Setting shake

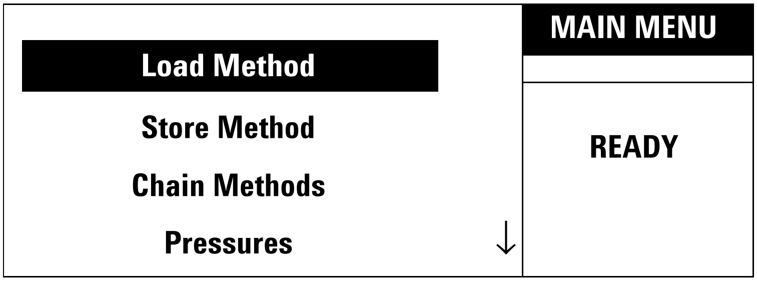
Shake specifies the agitation level of the oven carousel during vial equilibration. There are three shake settings: off, low, and high. Specify the level desired using the cursor keys followed by **Enter**.

Storing and Loading Methods

Storing a method

Store Method places the method in active memory into one of four storage locations for future use. To store the method currently in use:

1. Press **Menu**. This displays the following screen:



2. Use the cursor keys to highlight `Store Method`.
3. Enter the desired storage location {1–4} on the numeric keypad followed by **Enter**.

The Headspace Sampler stores the current method in the specified memory location. A stored method includes the following parameters:

- Zone Temps
- Event Times
- Vial Parameters
- The advanced functions for Oven Stabilization Time
- The advanced functions for Pressure Units
- The advanced functions for Multiple Headspace Extraction

CAUTION

Storing overwrites any method already in that location. There is no warning message that an overwrite is taking place.

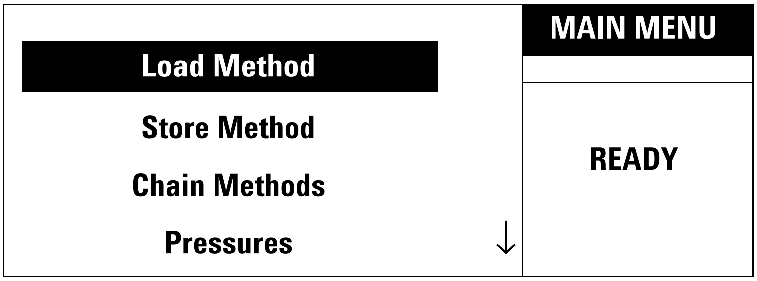
NOTE When the Headspace Sampler is first turned on after a loss of the battery–backed–up RAM, the default method (see page 74) is loaded into all four method–storage locations.

Loading a method

Use the Load Method function to retrieve a method from memory for use in the current analysis. Load Method can only be used to retrieve user defined methods. To load one of the five factory pre-set methods, see Stored Method on page 74.

To load a method:

1. Press **Menu**. This displays the following screen:



2. Use the cursor keys to highlight `Load Method`.
3. Enter the location desired {1–4} on the numeric keypad followed by Enter.

The method stored in the specified location is now available for current use.

Chaining Methods

Chain Methods allows two to four stored methods to be used sequentially to run groups of samples. It provides the user with some automation. Once the chain (or sequence) is programmed and the samples loaded, the sampler runs the sequence without further operator intervention. Method chaining is also a means of performing MHE.

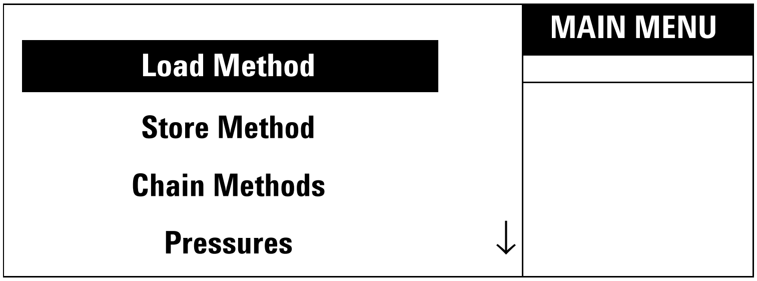
NOTE

If Parameter Increment is active, Chain Methods is inactive. Turn off Parameter Increment to use the method chain feature.

Activate a Method Chain

To activate a Method Chain:

1 Press **Menu**. This displays the following screen:



2 Use the cursor keys to highlight `Chain Method`. Press **Enter**

3 Key in the method sequence in the order to be run (up to four methods). Select from any of the available method numbers {1-4}.

4 Press **Enter** to activate the chain. There is no indication that the chain is enabled.

To confirm the method chain, highlight `Chain Methods` again and press **Enter**. This displays the current chain sequence. For example, a chain using methods 1, 2, and 3 displays the following:

Actual chain
1+2+3

Methods run in order from left to right on the display. This method sequence is active until you edit it or load a new method.

Deactivate a Method Chain

To deactivate a Method Chain:

1. Highlight `Chain Methods`. The display shows:

   Actual chain
   #+#+#+#

   where # is the number of a method.

2. Press **0** followed by **Enter**. The display shows:

   Actual chain
   Chain off

Using a Method Chain for MHE

Use Chain Methods to perform a MHE with multiple punctures and constant equilibration time for each extraction.

To run an MHE:

1. Key in the parameters for Zone Temps, Event Times and Vial Parameters for the method. See page 94.
2. Store this method in one of the four storage locations. See page 102.
3. Press the following:

   Chain Methods {X + X + X + X }

   where X is the location of the stored method.

7

Developing Methods

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This chapter describes the principals behind developing a method.

Developing Methods

This chapter discusses various parameters that can affect the sensitivity, precision, and accuracy of an analysis.

The tendency of a material to go into the gaseous phase is the partition coefficient, K, where C_c is the concentration of the analyte in the condensed phase (the sample matrix) and C_g is the concentration of the analyte in the gaseous phase (the headspace). The partition coefficient, K is related to the degree of solubility that the analyte has in the matrix. For example, benzene is not very soluble in water, and has a K of approximately seven. Ethanol, which is very soluble in water, has a K of 7000. A high value of K implies it is difficult for the analyte to leave the matrix and go into the headspace. See Figure 15 for an illustration of the particle coefficient.

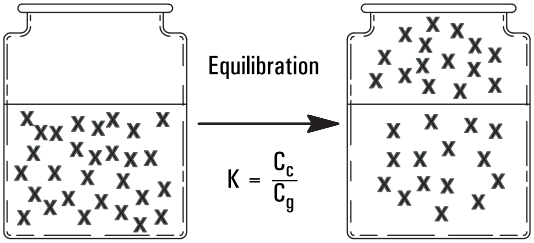

Figure 15 Illustration of partition coefficient

K is also very dependent on temperature, as demonstrated by the following equation:

$$\frac{dK}{dT} = \frac{1}{T^2}$$

Processes that reduce the value of K will increase the sensitivity of the headspace analysis. The following operations can be used to decrease K:

1. Add mineral salts such as NaCl or Na_2SO_4 to the matrix (aqueous samples).
2. Add another liquid to the matrix.
3. Increase the temperature.

Varying Oven Temperature

The oven temperature can have a profound effect on the concentration of analyte that passes into the headspace gas. In general, as the oven temperature increases, the amount of gas entering the headspace from the sample increases, increasing the pressure in the vial and delivering more analyte to the GC. This increases the sensitivity of the analysis.

Consider the following when choosing an oven temperature:

- Over-pressurization of the headspace vial could be dangerous.
- Unstable compounds could degrade at elevated temperatures.

There are two ways you can program the Headspace Sampler to increase oven temperature in steps:

- Use Chain Methods (see Chaining Methods on page 104) and set the oven temperature higher in each method in the chain
- Use Param Increment (see Parameter Increment on page 68).

NOTE

Do not set the oven temperature within 10 °C of the boiling point of any solvents in the sample, except in special cases.

Loop and Transfer Line temperatures should be set higher than the highest temperature the oven will reach in a programmed sequence.

Varying Event Times

Vial equilibration time

The time that the sample vial spends in the oven determines the amount of analyte in the headspace gas and the presence or absence of equilibrium. The Headspace Sampler can be programmed to analyze a series of samples to determine if equilibrium is reached.

To determine if equilibrium is reached, use Chain Methods (see Chaining Methods on page 104) to increase the Vial Equilibration Time for each method in the chain, or use Param Increment (see Parameter Increment on page 68) to increase the Vial Equilibration Time for each successive sample in the series. Then plot the peak area versus equilibration time for a certain peak. The system is at equilibrium when this curve flattens out. See Figure 16.

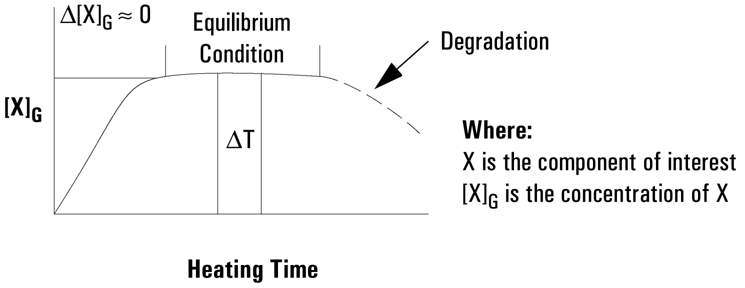

Figure 16 Equilibration curve

Loop fill time

Normally, set the loop fill time to 10 seconds.

A very short loop–fill time (one or two seconds) may help increase sensitivity in headspace analysis. The headspace gas purges the sample loop, but the vent valve is closed before the sample vial falls to atmospheric pressure. The loop is maintained at the vial’s still-elevated pressure. Because the loop pressure is greater, the analyte will be more concentrated and, consequently, more sample will be injected into the GC. A length of restrictive tubing can be attached to the Swagelok fitting labeled “vent” on the back of the Headspace Sampler to increase sensitivity.

Matrix Effects

The composition of the sample matrix can affect the amount of analyte that escapes into the headspace. Adding an inorganic salt to aqueous samples increases the concentration of organic molecules in the headspace by making them less soluble in the sample matrix, increasing sensitivity. For nonaqueous but water-miscible systems, dilute the sample with water. This decreases the solubility of most organic molecules in the sample and increases their concentration in the headspace.

Matrix effects are important when developing external standards. The composition of the matrix in the standard should closely resemble that of the sample, or accuracy suffers. The effect of the matrix on a standard can be compensated for by using the standard addition method or by quantifying results using MHE.

Changing the Sample Loop

Although the 1-mL sample loop is usually adequate, a larger sample injection for trace analysis might be desired. A 3-mL loop is available. See Changing the Sample Loop on page 134.

NOTE

A larger sample injection may broaden some peaks, especially on a capillary column. In general, higher carrier flow rates will be necessary.

Sample Size

A larger sample size may give greater sensitivity. Peak areas are often strongly influenced by the relative amount of the gas and condensed phases in the sample vial. Increasing the sample size may give corresponding increases in peak areas. If sensitivity is not an issue, small samples may be preferred because the required equilibration time is shorter.

Vial Pressurization and Time

When the sampler starts, it moves the first vial into the oven. Equilibration time begins. To shorten the required equilibration time, agitate the vial in the oven. See “Vial Parameters” on page 57.

Vial pressurization

With many liquid (for example, aqueous) samples, the pressure developed in the vial may be sufficient to ensure filling the valve loop without additional pressure. Some samples (for example, dry plastics) may not create enough head pressure, and do require more, in the method.

In general, 1.5–2 atmospheres pressure in the vial will be appropriate for filling the valve loop. It may be possible to make or buy a pressure gauge fitted with a needle that could be used to determine the actual vial pressure developed under your time and temperature conditions. Set the method vial pressurization at a slightly higher value (for example, 10–15 psi), if your measured pressure is not enough to ensure adequate loop filling.

CAUTION If the pressure setting is too low the vial pressurization flow path may become contaminated.

To optimize your selection of vial pressurization, run a series of vials using different pressures, and interpret the optimum conditions from peak areas versus vial pressurization.

NOTE Optimization is valid only for analysis of a specific sample at a specific oven temperature.

Vial pressurization time

Typical vial pressurizations times range from 10 to 30 seconds.

Optimizing Carrier Gas Flow

The Headspace Sampler carrier-gas flow rate should be set high enough to sweep the headspace sample out of the sample loop into the GC without causing peak broadening. For example, for a packed GC column that gives 15-second–wide peaks, flow would be at least 6-mL/min for a 1-mL loop. This will sweep out the loop in 10 seconds.

NOTE Some detectors, such as ECDs and TCDs, are more sensitive at lower detector flow rates.

8

Running a Sample

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  Setting carrier flow using EPC 123
  Setting vial pressurization flow using EPC 124

This chapter details the procedure for running a sample.

Running a Sample

To run a sample, perform the following:

1. Make sure the gas supplies have sufficient gas available for all of the analyses.
2. Set the flow and pressures for the Headspace Sampler. See page 121.
3. Set the GC conditions. See the GC operating manual.
4. Load the method or set the method chain. See Loading a method on page 103 or Activate a Method Chain on page 104.
5. Prepare the samples. See Sample Preparation on page 90.
6. Load the samples into the tray. See page 92.
7. When the GC and HS are ready, press **Start**.

Setting Conditions

Flow rates

The Headspace Sampler has two distinct methods of flow control. It can be used in MPC mode with mechanical pneumatic controls. It can also be used with a GC with EPC control. The procedure for setting flow rate differ for EPC and MPC.

Refer to the GC operating manual for details about its flow systems. See Figure 17 for the location of the Headspace Sampler pressure regulator and flow controller.

Make sure that the EPC/MPC toggle switches on the underside of the Headspace Sampler lid are set to the correct flow mode before proceeding. Use any combination of MPC and EPC flow control and pressure control. Refer to the Headspace Sampler installation manual for details.

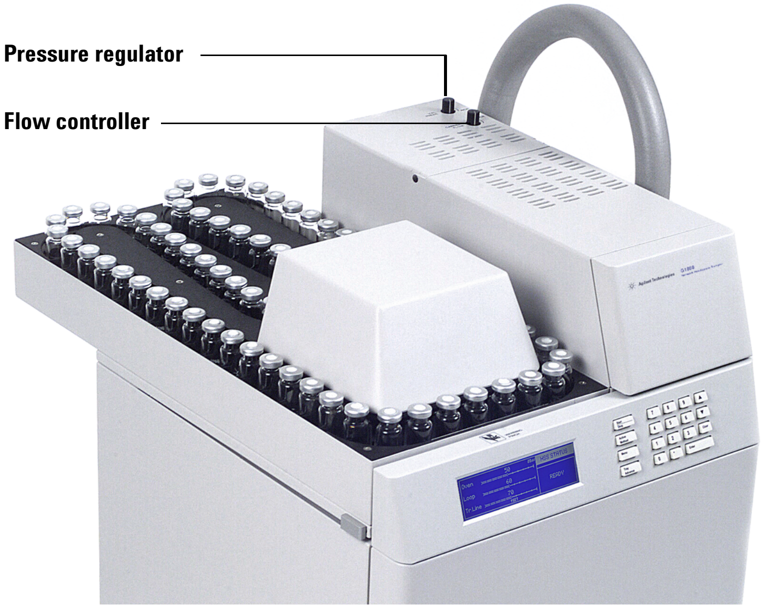

Figure 17 Location of flow controller and pressure regulator

Setting the carrier gas flow rate using MPC

Set the carrier gas flow as follows:

1. Set the GC parameters shown in per the method. Use a split ratio of 1:1.
2. Set the split flow to 4 mL/min. The GC total flow reads 11 mL/min.
3. Press **Menu** and choose `Pressure` from the menu. Use the cursor keys to scroll to `Carr. (PSI)`. The actual carrier gas pressure displays in the right side of the window. See page 62.
4. Connect an electronic flow meter to the GC split vent. Turn up the headspace carrier gas flow until the flow rate out the split vent equals the flow rate desired in the column. The split ratio is 1:1. This flow rate equals the column flow rate. See Figure 18. Remove the flow meter.

   Note that the GC and Headspace pressure readings should be similar. Record the headspace pressure reading—you can use it to set up future checkout runs.

CAUTION The added flow of the Headspace Sampler causes the flow calculations of the inlet to be incorrect. If the added flow and pressure exceeds the limits of the inlet, the GC may become “Not Ready.”

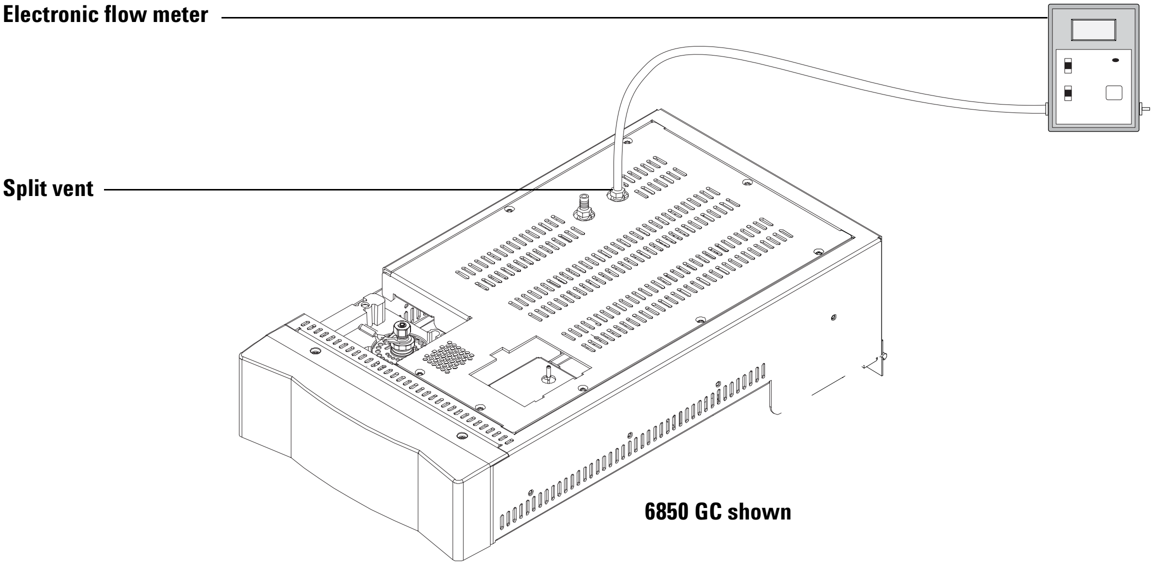

Figure 18 Measuring the split vent flow rate

Setting the vial pressurization gas pressure using MPC

Control the gas pressure used for vial pressurization using the pressure regulator. See Figure 17.

Set the vial pressurization gas higher than the pressure inside of the vial to prevent a backwards flow from contaminating the vial pressurization flow path. A setting of 10 to 20 psi is usually high enough.

To read the actual pressure value, press **Menu** and choose `Pressure` from the menu. The pressure displays in the right side of the window. See page 62.

Setting carrier flow using EPC

Set the carrier gas flow rate directly using the GC or GC data system. An aux EPC module is required when using the 6890 GC.

Setting vial pressurization flow using EPC

Set the vial pressure directly using the appropriate auxiliary channel in the GC. Auxiliary channels 3, 4, and 5 are the most common.

Set the vial pressurization gas higher than the pressure inside of the vial to prevent a backwards flow from contaminating the vial pressurization flow path. A setting of 10 to 20 psi is usually high enough.

9

Maintaining the Instrument

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This chapter describes the procedures for maintaining the instrument.

Maintenance Schedule

Table 8 lists routine maintenance activities that should reduce unplanned repairs and maintenance. The appropriate maintenance frequency for the instrument varies significantly depending on the sample matrix, solvents, temperatures, and sample throughput. Use Table 7 to determine the correct user type.

Table 7 General user types

Samples per day	Low boiling solvents (water, food, flavor analyses)	High boiling solvents (OVI, blood alcohol analysis)
≤ 70	User type 1	User type 3
> 70	User type 2	User type 4

Table 8 Recommended maintenance schedule

Maintenance activity	User type 1	User type 2	User type 3	User type 4
Replace sample probe	6 months	3 months	3 months	Every month
Steam clean	6 months	3 months	3 months	Every month
Replace loop and deactivated tubing	With PM	With PM	With PM	6 months
Check tray tension and alignment	12 months	6 months	12 months	6 months
PM for headspace and inlet	36 months	12 months	12 months	--
Extended PM for headspace	60 months	36 months	24 months	12 months

Common Replacement Parts and Consumables

Table 9 lists commonly used parts. Also refer to the Agilent catalog for consumables and supplies.

Table 9 Common Replacement Parts and Consumables

Description	Comment/Use	Part number
Tools		
Decapper, 20-mm		5181-1214
Crimper, 20-mm		9301-0720
Wrenches, 7 × 8 mm, Set of 2		1340407010
Wrenches, 8 × 10 mm, Set of 2		1340408000
Wrenches, 6 × 7 mm, Set of 2		1340406000
Hex L-wrench, 3 mm		1341203000
Hex L-wrench, 2.5 mm		1341002500
Leak test kit	For capping the transfer line and other connections	G1888-60701
Vial kits		
20-mL flat bottom Headspace crimp top vials, silver aluminum one piece crimp caps with safety feature, molded PTFE/butyl septa, 100/pk	General purpose	5182-0839
20-mL flat bottom Headspace crimp top vials, silver aluminum one piece crimp caps with safety feature, molded PTFE/silicone septa, 100/pk	Wider temperature range septa, need higher sensitivity.	5182-0840
20-mL flat bottom Headspace screw cap vials, clear color, 100/pk		5188-2753
20-mL flat bottom Headspace screw cap vials, amber color, 100/pk		5188-2754
Screw top caps, PTFE/silicone septa 100/pk		5188-2759

Table 9 Common Replacement Parts and Consumables (continued)

Description	Comment/Use	Part number
Sample loops		
1-mL deactivated steel		2321700003
3-mL deactivated steel		2321700004
Probes/unions		
Deactivated sample probe	Deactivated steel	2322700011
M6 union	Brass	2302533140
Zero dead volume union	Deactivated steel	2307230001
Union		2307232901
Transfer line needles and unions		
Transfer line needle, od 0.5, for split/splitless inlet	Deactivated steel	2322590004
Transfer line needle, od 0.7, for split/splitless inlet	Deactivated steel	2322590005
Strain relief septum nut		6410090050
Tubing		
Tubing, solenoids to 6-port valve	Deactivated steel	0410105017
Tube, sample probe to 6-port valve	Deactivated steel	1300502506
Manual		
Service CD-ROM		G1888-90008

Table 9 Common Replacement Parts and Consumables (continued)

Description	Comment/Use	Part number
Network Headspace Sampler User Information CD-ROM		G1888-90010
Site Preparation and Installation		
Operating		
Standards		
Headspace OQ/PV Standard kit:		5182-9733
1 ampoule of standard, 1 mL		
1 pkg of 5-mL micro-pipettes		
Material Safety Data Sheet		
Certificate of Analysis		
PM Kits		
G1888A PM kit with 1 mL loop		G18888-60702
G1888A PM kit with 3 mL loop		G18888-60703
G1888A enhanced PM kit		G18888-60704

Removing the Loop Cover

Remove the loop cover to gain access to most of the user serviceable parts. Follow the steps in Table 10.

Table 10 Removing the loop cover

Step	Action	Notes
1 Open the pneumatics cover.	**a** Raise the lid of the sampler. **b** Use a Phillips screwdriver to loosen the two screws at the front of the sampler cover shown below. **c** Lower the lid of the sampler. **d** Raise the pneumatics cover.	

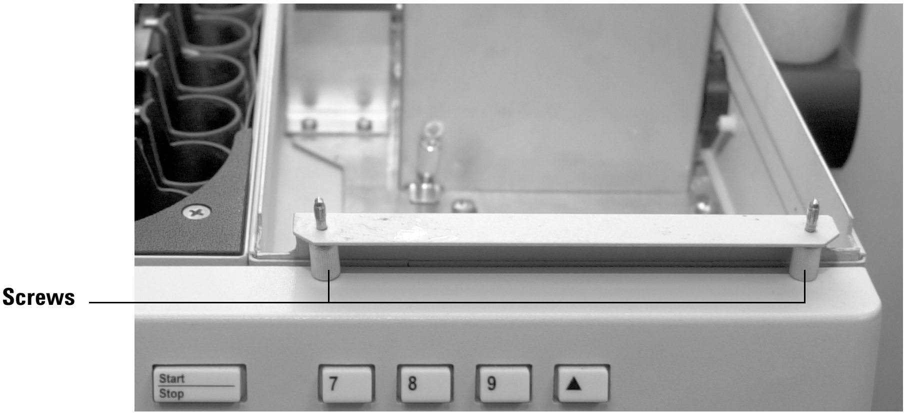
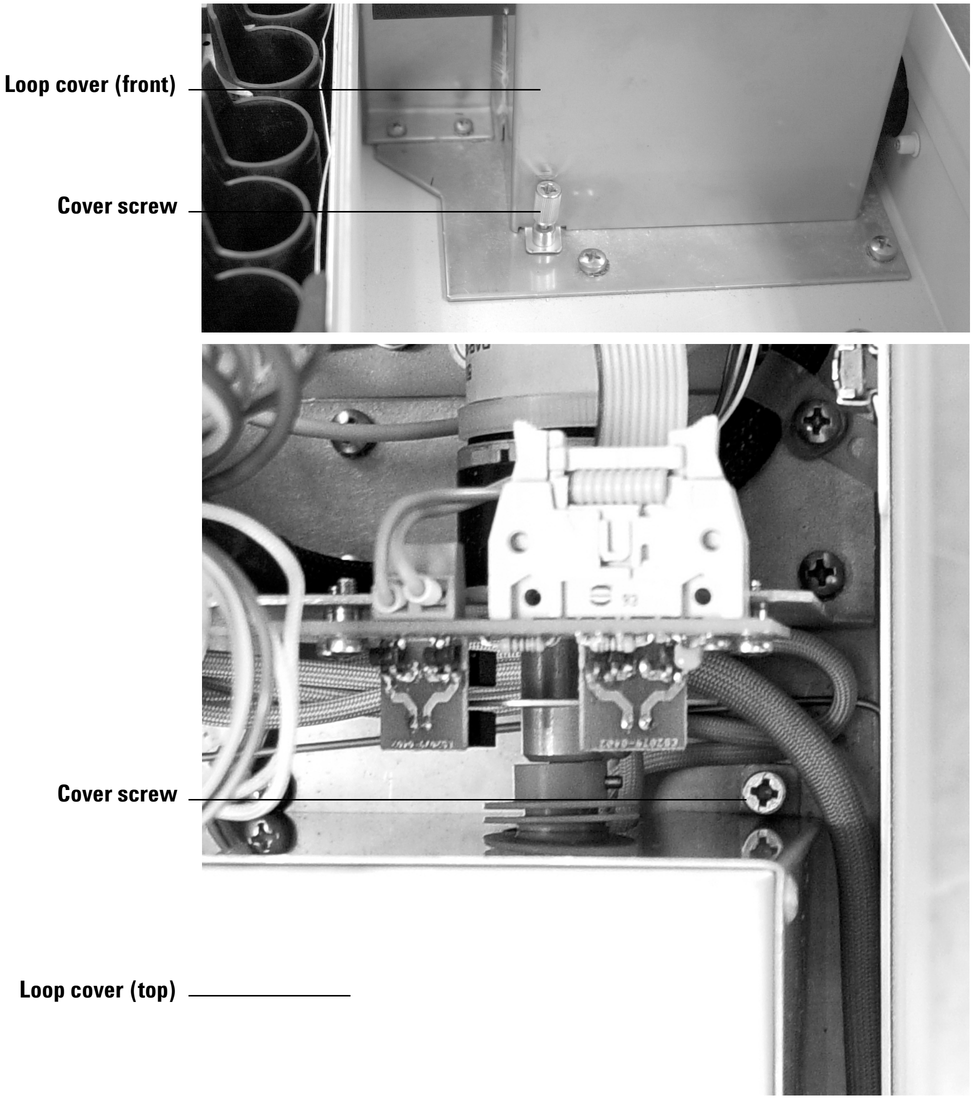


Table 10 Removing the loop cover (continued)

Step	Action	Notes
2 Remove the loop cover.	**a** Loosen the two screws on the loop cover. **b** Remove the cover.	


Loop cover (front)
Cover screw
Cover screw
Loop cover (top)

Changing the Sampling Probe

Follow the steps in Table 11 to change the sampling probe.

Table 11 Changing the sampling probe

Step	Action	Notes
1 Remove the loop cover. Follow the steps on page 130.		
2 Remove the heater block covering the sampling probe.	**a** Loosen the Phillips screw shown below. **b** Lift out the heater block.	

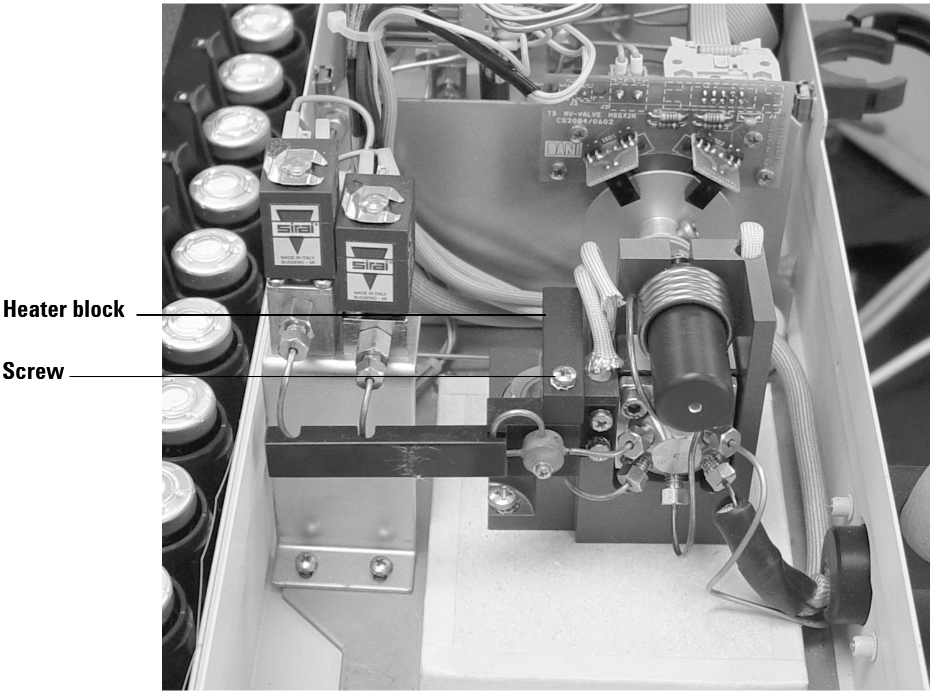
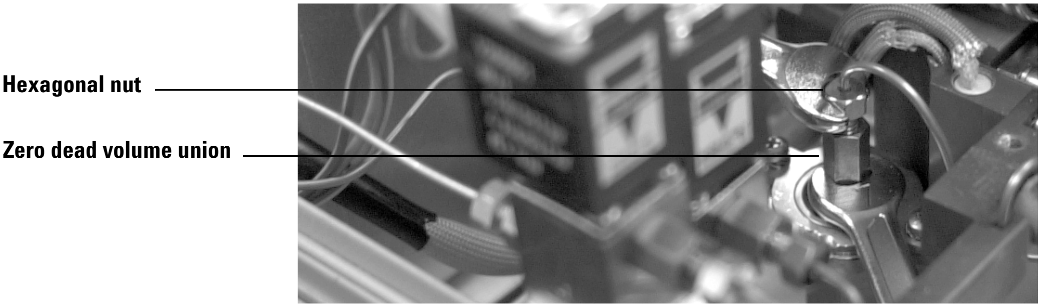
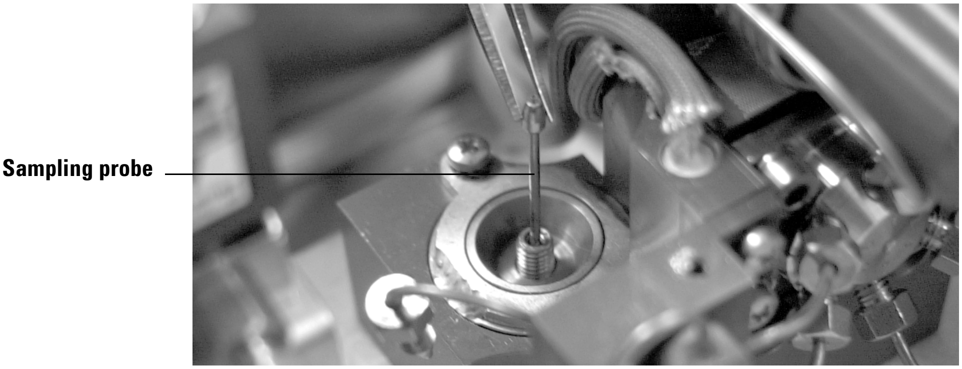


Table 11 Changing the sampling probe (continued)

Step	Action	Notes
3 Disconnect the plumbing from the sampling probe.	**a** Support the zero dead volume union with a 7-mm wrench and remove the nut with another 7-mm wrench. **b** Loosen the connection to port 5 on the 6-port valve. Lift the tube off of the sample probe and swivel out of the way. **c** Loosen and remove the zero dead volume union. See the figure below.	Do not bend any tubing when installing or the coating will break and compromise inertness.



Step	Action	Notes
4 Remove the sampling probe.	- Grab the sampling probe with tweezers and remove it from the unit.	



Step	Action	Notes
5 Insert a new sampling probe and reverse the previous steps to reassemble the Headspace Sampler.		- Tighten each fitting and perform a leak test before replacing the insulation and cover.

Changing the Sample Loop

Follow the steps in Table 12 to change the sample loop.

Table 12 Changing the sample loop

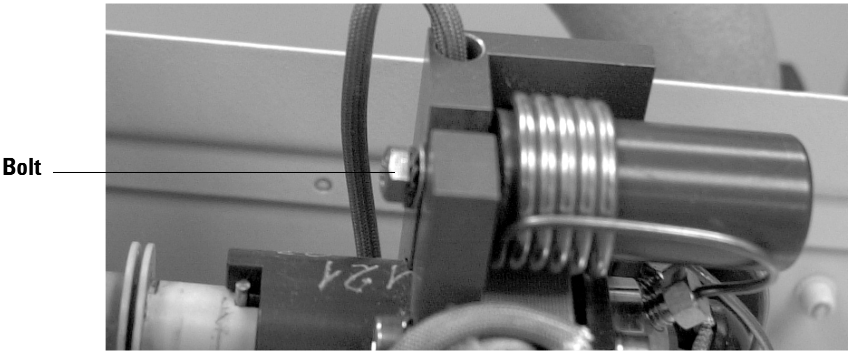
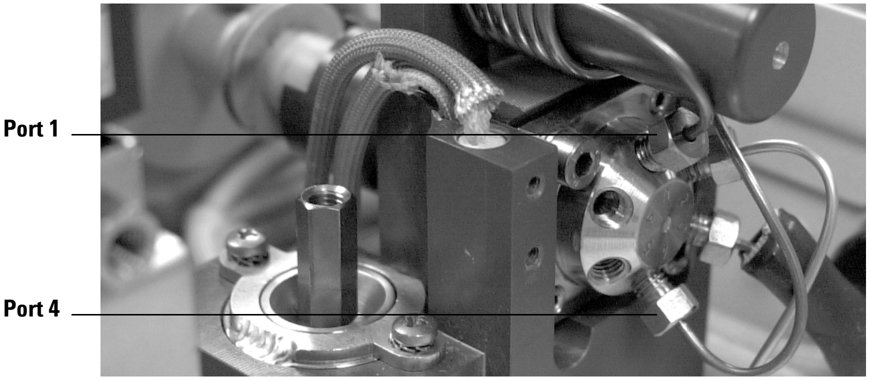
Step	Action	Notes
1 Remove the loop cover. Follow the steps on page 130.		
2 Loosen the bolt holding the back of the loop mandrel to the manifold body.	- Use a 7-mm wrench to loosen the bolt shown below.	
		
3 Remove the plumbing from the six-port valve.	- Use a 7-mm wrench to loosen the two nuts connecting the sample loop to ports 1 and 4 of the six-port valve. See the figure below.	

Table 12 Changing the sample loop (continued)

Step	Action	Notes
		
4 Exchange the sample loop.	**a** Slide the loop off the mandrel. **b** Slide the new sample loop onto the mandrel.	• When installing a deactivated sample loop, take care not to bend the loop when installing it or you will break the coating and compromise inertness.
5 Reverse the steps given above to reassemble the Headspace Sampler.		• Perform a leak test on fittings before replacing the insulation and manifold cover.

Replacing the Tubing Assembly

Follow the steps in Table 13 to change the tubing assembly.

Table 13 Replacing the tubing assembly

Step	Action	Notes
1 Remove the loop cover. Follow the steps on page 130.		
2 Remove the heater block covering the sampling probe and tubing assembly.	**a** Loosen the Phillips screw shown below. **b** Lift out the heater block.	

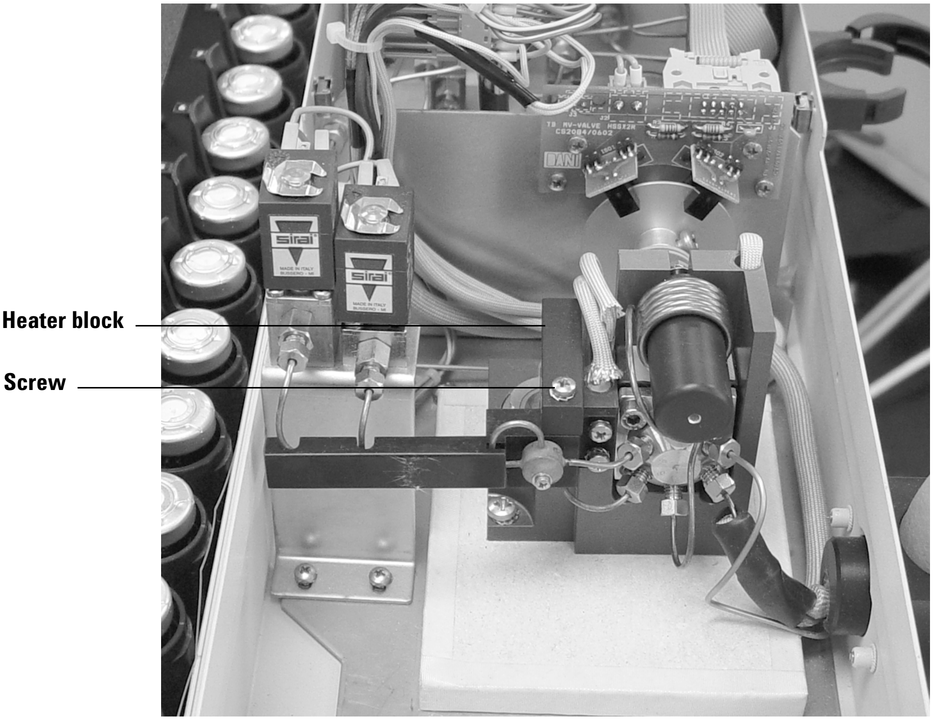
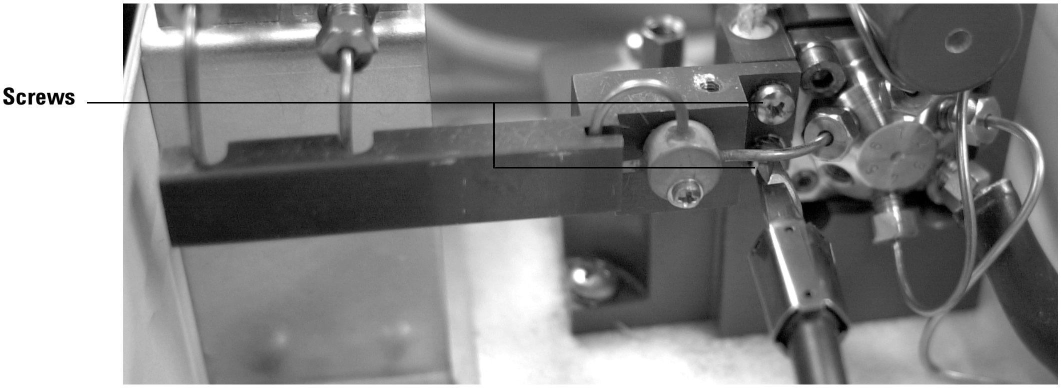

Heater block
Screw
sirai
sirai
MADE IN ITALY
DANI
TB MV-VALVE HSSX2K
CS2084/0602

Table 13 Replacing the tubing assembly (continued)

Step	Action	Notes
3 Remove the two screws.	- Remove the two Phillips head screws shown below.	



Step	Action	Notes
4 Disconnect the plumbing	**a** Disconnect the solenoid valves. Use an 8-mm wrench to secure the connection at the solenoid fitting and a 7-mm wrench to turn the connection on the tubing. **b** Disconnect the fitting from port 6 of the 6-port valve. Use a 7-mm wrench.	

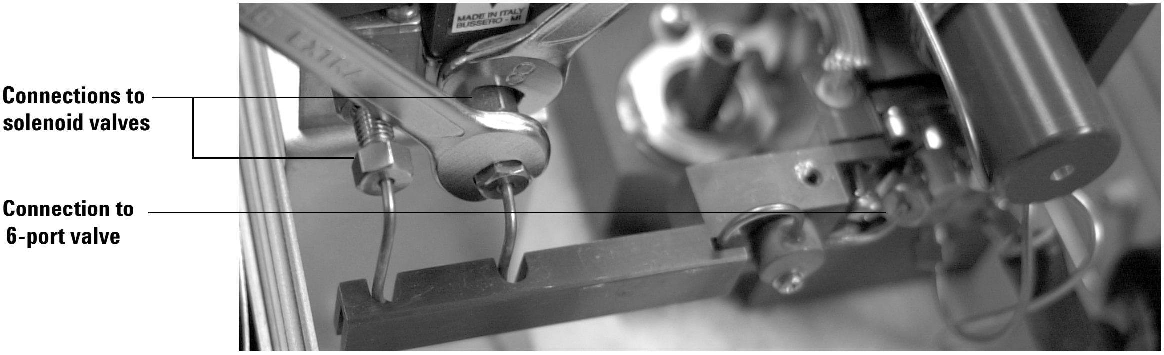

Connections to solenoid valves
Connection to 6-port valve

Table 13 Replacing the tubing assembly (continued)

Step	Action	Notes
5 Replace the tubing assembly	• Remove the old assembly and place the new assembly in the Headspace Sampler.	Take care not to bend the tubing when installing it or you will break the coating and compromise inertness.
6 Reverse the steps given above to reassemble the Headspace Sampler.		• Perform a leak test on fittings before replacing the insulation and manifold cover.

Checking Alignments

Reliable vial delivery depends on these key adjustments:

- Tray chain tension.
- Tray position 1 sensor alignment.
- Tray/shutter motor group alignment.
- Carousel alignment.
- Shutter alignment.

Use the following procedures to check for alignment problems.

Checking the tray chain tension

If the tray is not properly tensioned the chain links can rotate around their hinges and prevent the vial from centering over the shutter hole during vial delivery. The result is a Vial Not Dropped error.

Perform the following visual check to verify the chain tension:

1. Push the chain to the left hand standoff.
2. Verify that the tray chain resists and springs back after releasing it.

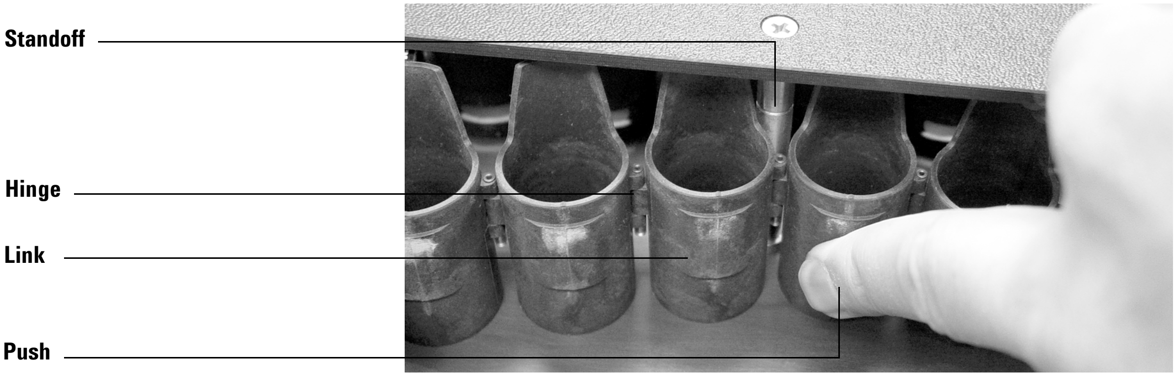


Figure 19 Tray chain alignment

If the chain tension is not correct, call Agilent service.

Checking the position 1 sensor alignment

If the position 1 sensor is not aligned the chain does not line up over the oven lift hole. The result is a vial not dropped error. Perform the following visual check:

1. Use tray advance (see page 50) and move the chain to position 35 and back to position 1.
2. Use the advanced function Manual Operation (see page 72) to home the tray. Press **Menu** and select `Advanced Functions`. On the keypad, press **5** and **Enter**. Scroll to `Tray`. Press **5** on the keypad.
3. Verify that the detail on the tray chain link lines up with the dimple on the tray wall. See Figure 20. If it does not align call an Agilent service representative.

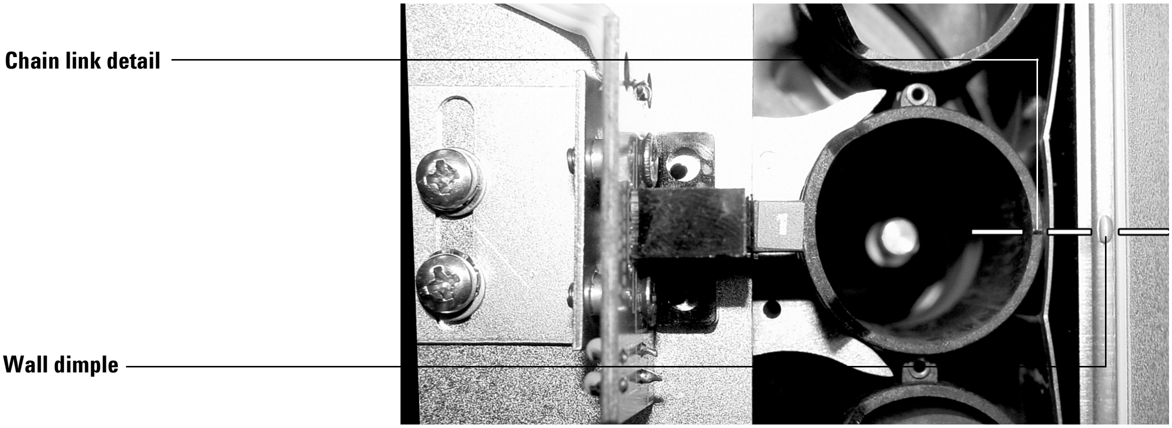


Figure 20 Tray chain alignment

Checking the tray/shutter motor group alignment

If the tray/shutter motor group is not aligned, the chain or the shutter hole may not be centered over the hole in the lid. See Figure 21. The result is a Vial Not Dropped error. If the group is too far to the right, the shutter will not turn properly. The result is usually a shutter error. Perform the following visual check to verify the alignment:

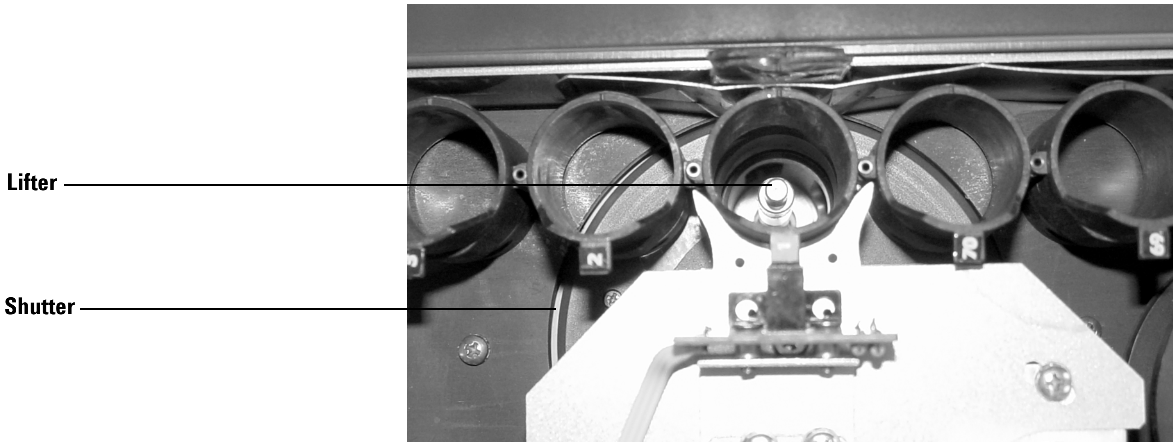

Lifter
Shutter

Figure 21 Motor group alignment

1 Visually check that the shutter is not touching the edge of the tray base. See Figure 22.

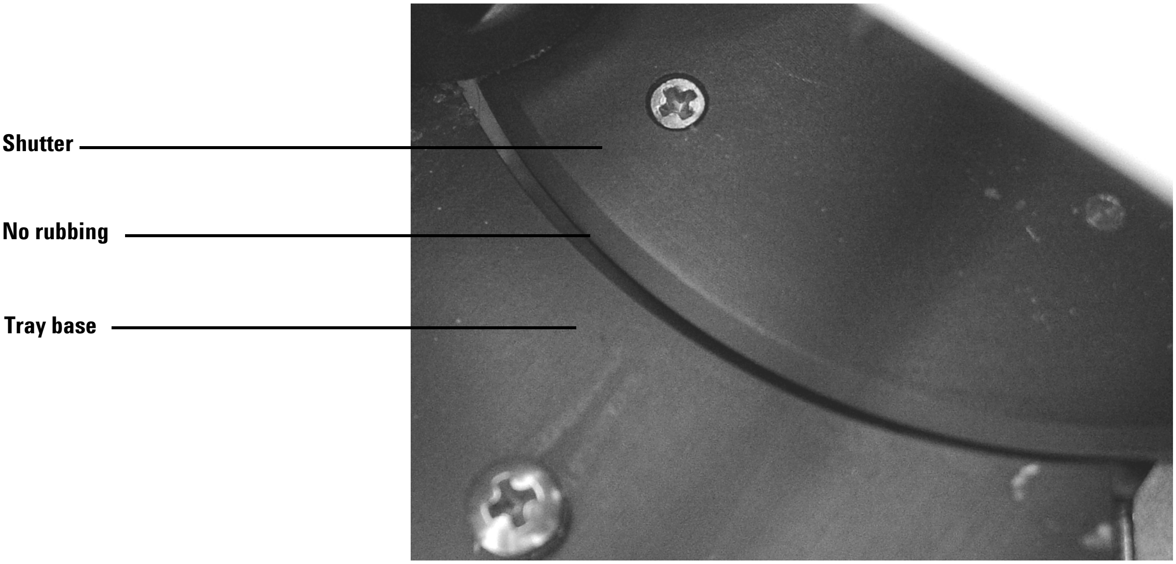

Shutter
No rubbing
Tray base

Figure 22 Shutter alignment

2. Power on the instrument. Watch the self-test and listen for the distinctive clatter of the shutter
3. Use Advanced Function 5 Manual Operation (see page 72) to check the shutter alignment. Scroll to `Shutter` with the cursor keys. Use the **1** key to move the shutter to mid and open positions. Use the **0** key to move the shutter back to mid and close positions.

If the shutter is not properly aligned, call Agilent service.

Aligning the carousel

If the carousel is not properly aligned, the hole in the lid does not line up with the cylindrical vial holder in the carousel. See Figure 23. The result is a Vial Not Dropped error. If the alignment is off by more that a few millimeters, the holes in the carousel for the lifters do not line up. The result is lifter errors.

WARNING **The surfaces of the oven, carousel, and oven lid may be hot and could burn you. Before beginning, set the oven and loop temperatures off. Allow the oven to cool to room temperature.**

Perform the following visual check:

1. Open and lock the lid.
2. Locate carousel position 1. It is marked with one small indentation.
3. Use the advanced function Manual Operation (see page 72) to home the carousel. Press **Menu** and select `Advanced Functions`. On the keypad, press **5** and **Enter**. Scroll to `Carousel`. Press **1**on the keypad to move the carousel one position in the clockwise direction. Press **0** to move the carousel one position in the counterclockwise direction.
4. Scroll to `Shutter`. Press **1** to open the shutter.
5. Scroll to `Lifter tray`. Press **1** to raise the lifter.
6. Verify that the lifter is centered in the hole of the carousel.
7. Reset using Advanced Function 10 or power cycle the sampler.

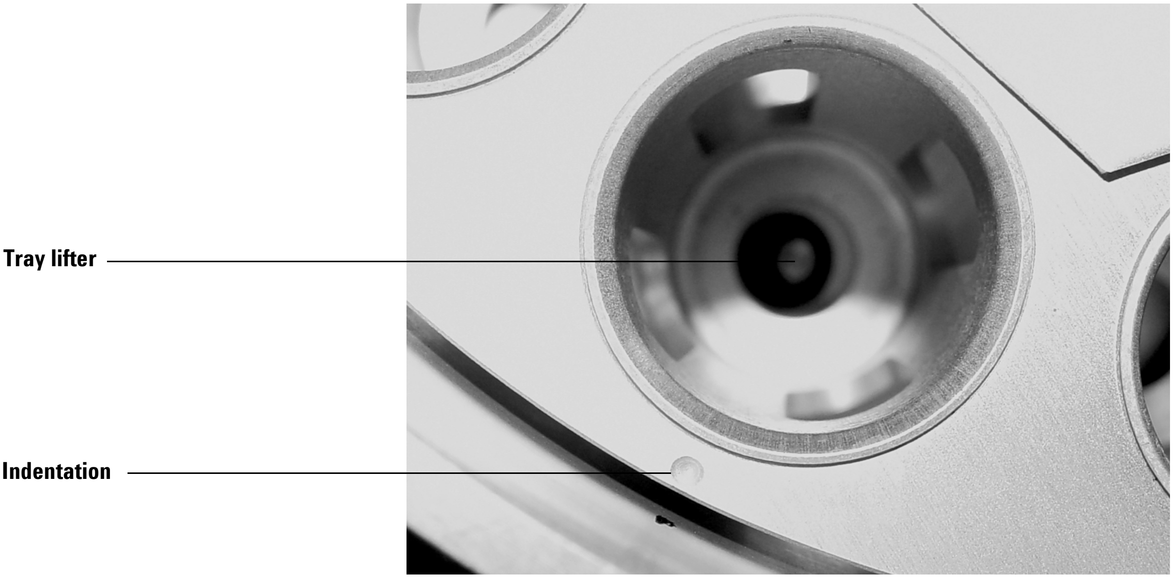

Tray lifter
Indentation

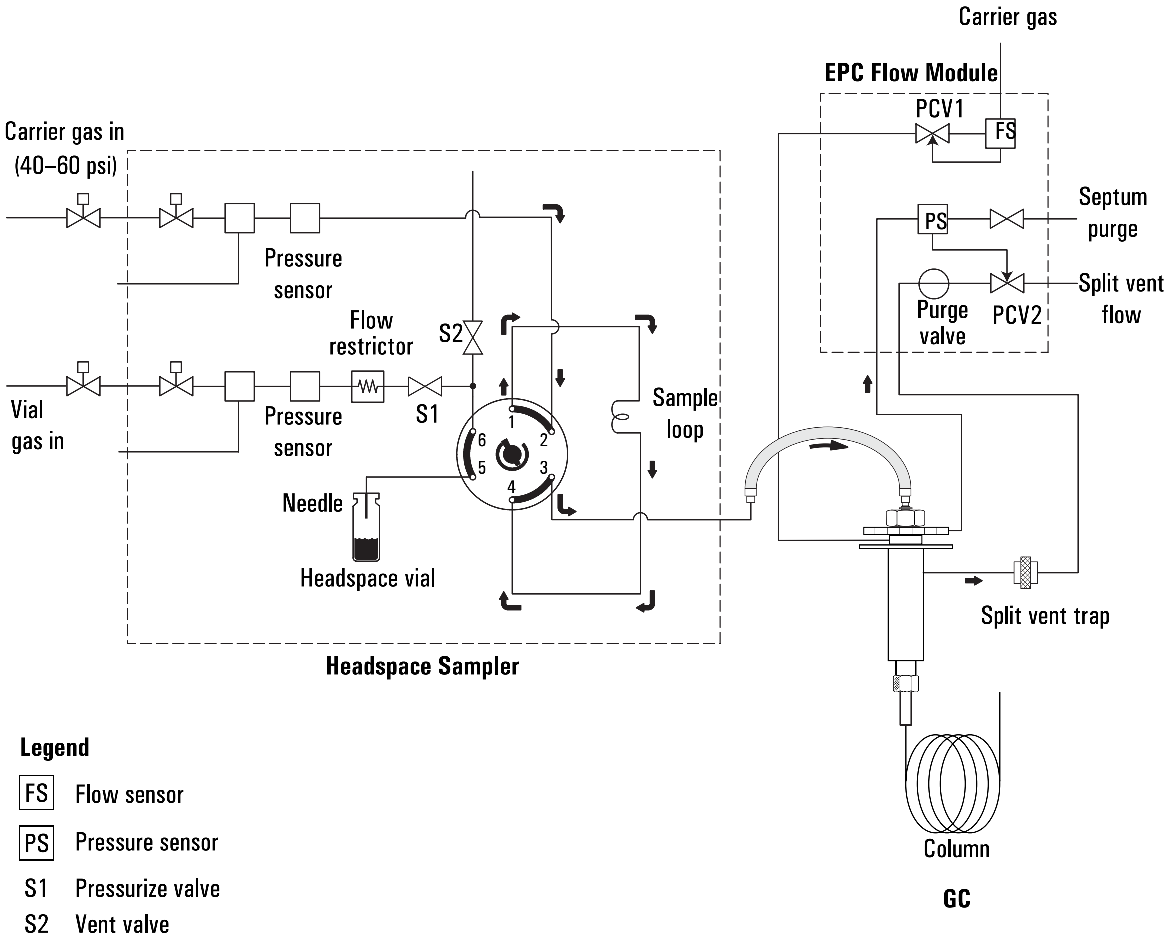
Figure 23 Carousel alignment

If the elevator rod and bushing do not line up exactly under the hole in the carousel cylinder, call Agilent service.

Leak Testing: General Information

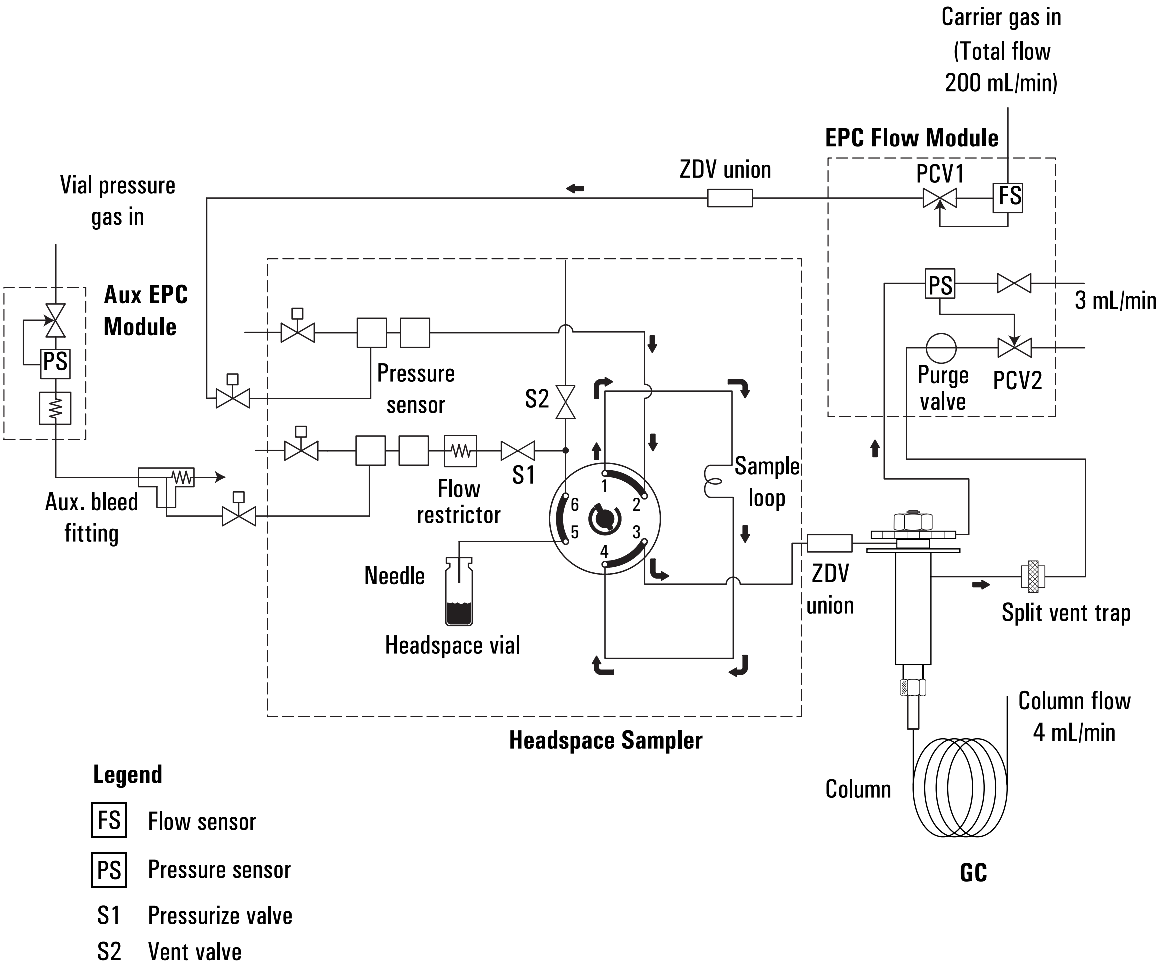
Refer to Figure 24 and Figure 25, which show the flow paths for all gases used in EPC and MPC headspace installations.

Chromatographic symptoms such as loss of sensitivity can result from leaks and restrictions. Use the following set of tests for verifying leaks and restrictions within the Headspace Sampler. Verify that the GC inlet is leak free before checking for leaks within the Headspace Sampler.


Carrier gas
EPC Flow Module
PCV1
FS
PS
Septum purge
Split vent flow
Purge valve
PCV2
Carrier gas in (40–60 psi)
Pressure sensor
Flow restrictor
S2
S1
Vial gas in
Pressure sensor
Sample loop
1
2
3
4
5
6
Needle
Headspace vial
Headspace Sampler
Split vent trap
Column
GC
Legend
FS Flow sensor
PS Pressure sensor
S1 Pressurize valve
S2 Vent valve

Flow paths before GC prep-run

Figure 24 Flow paths for an MPC G1888 to GC installation


Carrier gas in
(Total flow
200 mL/min)
EPC Flow Module
PCV1
FS
ZDV union
Vial pressure
gas in
Aux EPC
Module
PS
PS
3 mL/min
Purge
valve
PCV2
Pressure
sensor
S2
Sample
loop
Flow
restrictor
S1
1
2
3
4
5
6
Aux. bleed
fitting
Needle
Headspace vial
ZDV
union
Split vent trap
Headspace Sampler
Column flow
4 mL/min
Column
GC
Legend
FS Flow sensor
PS Pressure sensor
S1 Pressurize valve
S2 Vent valve

Flow paths before GC prep-run

Figure 25 Flow paths for an EPC G1888 to GC installation

After maintenance or repair, leaks are most often found where fittings were disconnected and reconnected. These areas include:

- Transfer line connections to the inlet such as needle and septum, zero dead volume union, and volatiles inlet.
- Connections to the 6-port sampling valve.
- Zero dead volume union above the sampling probe.

In some applications, residues and aggressive solvents can cause damage to the 6-port valve and restrictions in the vent tube and solenoid vent valve.

Leak Test

A leak test checks for the following:

- Restrictions in the flow path associated with filling the loop
- Leaks in the carrier flow path
- Leaks in the vial pressurization flow path
- Leaks across the 6-port sampling valve

The most common leaks are at the connections of the transfer line, external plumbing, and sample probe union.

Required tools

- Beaker of water
- 20-mL capped vial
- Leak test kit G1888-60701
- 7 × 8-mm wrenches
- This test can be facilitated using the Advanced Function 5 diagnostic tool located on the CD ROM.

Setup

1. Attach the 1/8-inch od tubing to the vent with a Swagelok connection. Finger tighten the connection.
2. Place the free end of the tube in a beaker of water.
3. If needed, install a shut-off valve between the gas source and the bulkhead fitting for carrier and vial pressurization gas. Install the valves on the back of the Headspace Sampler between the bulkhead fittings and the gas source.
4. For EPC setups, add a T-fitting connecting the shutoff valves on the carrier gas and vial pressurization gas inlets.
5. Change the pressure units to PSI. This setting has better resolution than kPa or Bar. See page 73.
6. Set the source pressure to 30 PSI.

7. Disconnect the transfer line from the GC inlet. If a transfer line needle is used, plug off the end with a septa. If there is no needle, plug off the end with a union and plug.
8. Place an empty 20-mL capped vial in tray position 1. Make sure the Headspace Sampler is set for 20-mL vials. See page 80.
9. Place the aluminum leak test vial in tray position 2. Place the end of the vial labeled “G1888” facing up.
10. Press the **Menu** key. Select `Advanced Functions`. Choose menu item 18 `Leak Test` and press **Enter**. Press **Enter** again to begin the test.

Running the restriction test

The Headspace Sampler loads the vial in tray position 1 into the oven carousel and raises it onto the sampling probe. The vial remains on the probe for the this part of the test.

1. The pressurization valve opens and pressurizes the vial for 0.2 minutes. After 0.2 minutes, the pressurization valve closes. The vent valve opens for 0.3 minutes. The vented air flows through ports 1, 3, 5, and 6 of the sampling valve.

   Watch the beaker of water. The valve clicks and bubbles emerge from the tube in the water. Normal behavior is a burst of bubbles that slows and stops after 4-5 seconds.

2. The pressurization valve opens and pressurizes the vial for 0.2 minutes. After 0.2 minutes, the pressurization valve closes. The vent valve opens for 0.3 minutes. The vented air flows through ports 5 and 6 of the sampling valve.

   The instrument switches the 6-port valve to the inject position. Watch the beaker of water. The valve clicks and bubbles emerge from the tube in the water. Normal behavior is a burst of bubbles that slows and stops after 3-4 seconds.

3. At the end of the restriction test, the instrument returns the empty glass vial to tray position 1. The Headspace Sampler sets the valves in standby mode and loads the aluminum leak test vial from tray position 2.

If there are are no bubbles or a slow stream of bubbles, call an Agilent service representative. The Headspace Sampler may have the following problems:

- Plugged or restricted vent flow paths
- Plugged or restricted valves
- Leak in the vent flow path

Pressure decay test

Overview

Each pressure decay test has four steps:

1. Pressurize the flow path long enough for the pressure to equalize across manual pneumatic controls and the flow restrictor.
2. Seal the flow path.
3. Monitor the pressure for 5 minutes.
4. Check the results. The pressure should decay less than 2 psi over 5 minutes.

The pressure decay test is normally done twice:

- Once with the sampling loop in the vial pressurization flowpath (standby mode)
- Once with the sampling loop in the carrier flow path (inject mode).

An optional pressure decay test checks for leaks across the 6-port valve. This optional test requires up to four additional pressure decay tests.

Pressure decay test part 1: Standby mode

The first pressure decay test checks for leaks in standby mode. Perform the following steps to run the pressure decay test:

1. Pressurize the flow paths for at least 5 minutes.
2. Turn off the shutoff valves at the back of the instrument.
3. Print Table 14. Write the time and initial pressure values in the table.

Table 14 Pressure decay test part 1

	Final time	**Initial time**	**Difference**
Time			5 minutes
Flow path	**Initial pressure**	**Final pressure**	
Carrier			
Vial pressurization			

4. Allow 5 minutes to pass. Record the final time and pressure values in Table 14.

A pressure decay of more than 2 psi over 5 minutes indicates a leak. Be sure that the leak is not at the transfer line.

- A leak in the carrier flow path indicates a possible leak at port 2 or 3 of the 6 port valve.
- A leak in the vial pressurization flowpath indicates a possible leak at port 1, 4, 5, or 6 of the 6 port valve.

Fix any leaks before continuing the leak test. Once all of the fittings have been tightened, start the pressure decay test again. If the pressure decay test still shows a leak or if the leak could not be fixed or located, call an Agilent service representative.

Pressure decay test part 2: 6-port valve standby mode (optional)

This section consists of two pressure decay tests. The tests check for internal cross leaks in the 6-port valve in standby mode. To skip this section, proceed to Pressure decay test part 3: Inject mode.

1. Loosen or disconnect the carrier gas connection at the transfer line. The Carrier (PSI) reading should drop to almost 0 PSI within 1 minute.
2. Print Table 15. Record the time and initial vial pressure in the table.

Table 15 Pressure decay test part 2A

	Final time	**Initial time**	**Difference**
Time			5 minutes
Flow path	**Initial pressure**	**Final pressure**	
Vial pressurization			

3. Allow 5 minutes to pass. Record the final time and pressure values in Table 15. A pressure decay of more than 2 psi over 5 minutes indicates a leak. If there is a leak, call an Agilent service representative. If there is no leak, proceed to step 4.
4. Tighten or reconnect the carrier gas connection at the Transfer line. Open the shutoff valve to pressurize the carrier flow path. The Carrier (PSI) should recharge within 1 minute.
5. Open the lid. The sample probe disengages from the leak test vial. The Vial (PSI) should drop within a minute to almost 0 psi.
6. Turn off the carrier shutoff valve at the back of the instrument.
7. Print Table 16. Write the time and initial pressure values in the table.

Table 16 Pressure decay test part 2B

	Final time	Initial time	Difference
Time			5 minutes
Flow path	**Initial pressure**	**Final pressure**	
Carrier			

8 Allow 5 minutes to pass. Record the final time and pressure values in Table 16. Carefully close the lid of the sampler so the sample probe enters the leak test vial.

A pressure decay of more than 2 psi over 5 minutes indicates a leak. If there is a leak, call an Agilent service representative. Fix any leaks before continuing the leak test.

Pressure decay test part 3: Inject mode

This pressure decay test checks for leaks in inject mode. Perform the following steps to run the pressure decay test:

1. Open the shutoff valves on the back of the Headspace Sampler.
2. Wait 5 minutes for the flow paths to pressurize and equilibrate.
3. Turn off the shutoff valves on the back of the Headspace Sampler.
4. Print Table 17. Record the time and initial pressure values in the table.

Table 17 Pressure decay test part 3

	Final time	Initial time	Difference
Time			5 minutes
Flow path	**Initial pressure**	**Final pressure**	
Carrier			
Vial pressurization			

5 Allow 5 minutes to pass. Record the final time and pressure values in Table 17.

A pressure decay of more than 2 psi over 5 minutes indicates a leak. Be sure that the leak is not at the transfer line.

- A leak in the carrier flow path indicates a possible leak at port 1, 2, 3, or 4 of the 6 port valve.
- A leak in the vial pressurization flowpath indicates a possible leak at port 5 or 6 of the 6 port valve.

Fix any leaks before continuing the leak test. Once all of the fittings have been tightened, start the pressure decay test again. If the pressure decay test still shows a leak or if the leak could not be fixed or located, call an Agilent service representative. To finish the leak test with the optional check of the 6-port valve, proceed to part 4. Otherwise, press **Clear** to end the leak test.

Pressure decay test part 4: 6-port valve inject mode (optional)

This section consists of two pressure decay tests. The tests check for internal cross leaks in the 6-port valve in inject mode. This section is optional.

1 Loosen or disconnect the carrier gas connection at the transfer line. The Carrier (PSI) should drop to almost 0 psi within 1 minute.

2 Print Table 18. Record the time and initial pressure values in the table.

Table 18 Pressure decay test part 4A

	Final time	Initial time	Difference
Time			5 minutes
Flow path	**Initial pressure**	**Final pressure**	
Vial pressurization			

3 Allow 5 minutes to pass. Record the final time and pressure values in Table 18. A pressure decay of more than 2 psi over 5 minutes indicates a leak. If there is a leak, call an Agilent service representative. If there is no leak, proceed to step 4.

4 Tighten or reconnect the carrier gas connection at the transfer line. Open the shutoff valve to pressurize the carrier flow path. The Carrier (PSI) should recharge within a minute.

5 Open the lid. The sample probe disengages from the leak test vial. The Vial (PSI) should drop to almost 0 psi within 1 minute.

6 Turn off the carrier shutoff valve at the back of the Headspace Sampler.

7 Print Table 19. Record the time and initial pressure values in the table.

Table 19 Pressure decay test part 4B

	Final time	**Initial time**	**Difference**
Time			5 minutes
Flow path	**Initial pressure**	**Final pressure**	
Carrier			

8 Allow 5 minutes to pass. Record the final time and pressure values in Table 19. Carefully close the lid of the sampler so the sample probe enters the leak test vial.

A pressure decay of more than 2 psi over 5 minutes indicates a leak. If there is a leak, call an Agilent service representative.

Press the **Clear** key to end the leak test.

Zone Calibration

Use Zone Calibration to create a custom temperature offset. In most cases a custom offset is not needed. However, a custom offset may be useful in the following situations:

- The laboratory has an ambient temperature outside of the Headspace Sampler's recommended operating temperatures.
- The method requires using the Headspace Sampler's maximum or minimum temperature settings.
- A particular sample matrix and equilibration time requires greater accuracy for oven temperature and/or loop temperature.

The calibration process may take days to complete. Once completed, the custom offsets are stored in memory on the main processor PCA. If this PCA fails, the custom offset cannot be restored. Do the following as a precaution:

- Keep a copy of the temperature readings taken during calibration so they can be re-entered in the event of a failed main processor PCA.
- Have an Agilent service representative backup the custom setting data with the diagnostic utility.

Print Table 20 and use it to record zone calibration data.

Table 20 Zone calibration data

Setpoint for oven	Oven measurement	Setpoint for Loop	Loop measurement
50 °C		65 °C	
80 °C		95 °C	
110 °C		125 °C	
140 °C		165 °C	
170 °C		185 °C	
200 °C		215 °C	
230 °C		245 °C	
230 °C		245 °C	

Recommended tools

You will need the tools listed in Table 21 to complete the zone calibration procedure.

Table 21 Recommended tools

Qty	Description	Notes
1	20-mL sample vial and cap	Recommend vial part number 5182-0837 and cap and septum part number 5183-4477.
1	No.1 Phillips head screw driver	Used for accessing the loop heated zone.
1	High temperature tape	Used to secure the probe lead from the oven.
1	Timer	Used for tracking the equilibration time between the zone reaching the set point and the measurement.
1	Digital thermometer	Recommend 2 inputs with accuracy of ±0.2 °C.
2	Type K thermocouple bead probe	Able to function between 0 °C and 260 °C with an accuracy of ±1.1 °C

Procedure

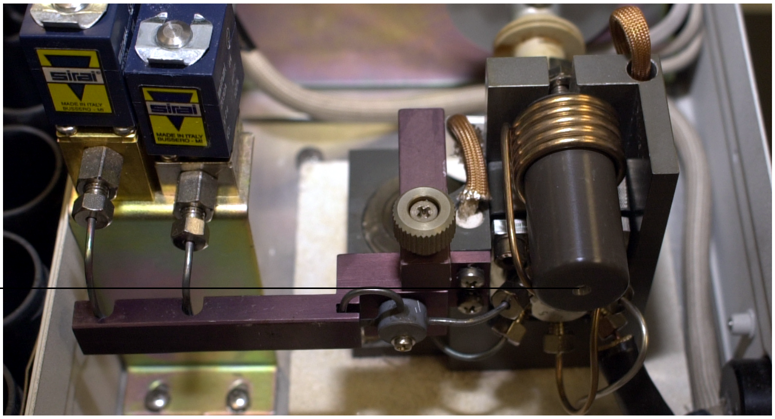
The procedure consists of three tasks:

1. Collect the temperature measurements
2. Enter the measurements into the Headspace Sampler
3. Store the custom offset

Follow the procedure in Table 22.

Table 22 Collect the temperature measurements

Step	Action	Notes
1 Prepare the vial.	**a** Fill a 20-mL vial with 10 mL of dry sand **b** Cap the vial (see page 90) and poke a small hole in the middle of the septum. **c** Push the K-type probe through the hole and into the sand. Make sure the probe is not touching the wall of the vial.	
2 Start the calibration process from the Zones Calibration screen.	**a** Navigate to Advanced Function 13. See page 81. **b** Use the cursor keys to highlight `Oven Calibration` from the Zones Calibration screen. Press **Enter**.	
3 Place the temperature probes.	**a** Lift the lid of the Headspace Sampler and place the vial with temperature probe in carousel position 1. **b** Remove the manifold cover over the sampling loop. See page 130. **c** Place the second K-type probe into the hole in the heater block that holds the sample loop. See below. **d** Replace the manifold cover and tighten screws.	• Route the sample loop probe lead along the transfer line to avoid pinching. • Push the probe to the back of the hole in the sample loop heater block.



Place temperature probe here

Table 22 Collect the temperature measurements (continued)

Step	Action	Notes
4 Begin the calibration process.	**a** Attach the K-type probes to the digital thermometer **b** Press **Enter** on the Headspace Sampler to continue.	
5 Record temperature readings.	**a** Allow 1 hour for the Headspace Sampler to reach the setpoint shown. **b** Record the temperature shown on the digital thermometer. **c** Press the down arrow key to move to the next temperature setpoint. Repeat until each setpoint has been recorded.	• To repeat a measurement at a lower setpoint, press the up arrow key until the setpoint is displayed on the screen. Open the lid of the oven to allow cooling. • Record the temperature reading in Table 20.
6 Enter temperature measurements.	**a** The Headspace Sampler displays a table showing the temperature setpoints. Use the keypad to enter the measured values under each setpoint. **b** Press the down arrow to move to the next setpoint. Repeat until each measurement is entered.	
7 Calculate offset.	**a** After each of the 14 temperature settings is entered, the HS asks "Ready to process custom temperature offset?" **b** Press **Enter** to complete. Press **Clear** to go back.	

Fuse locations

Refer to Figure 26 and Table 23 below for fuse locations and part numbers.

WARNING **Fuse replacement should be performed only by qualified service personnel.**

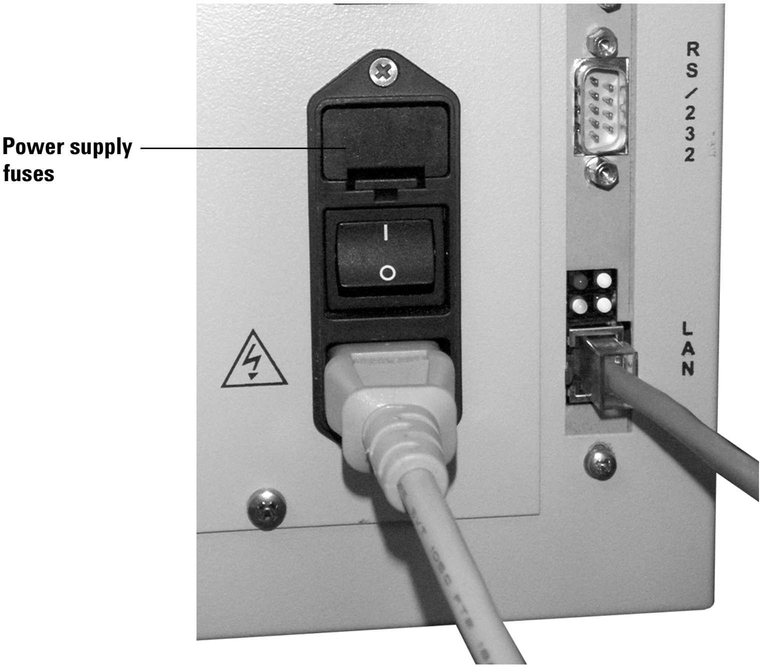

Power supply fuses
RS/232
LAN
I
O

Figure 26 Fuse locations on the power supply

Table 23 Fuses

Fuse designation	Location	Fuse rating and type
F2, F3	Power line module	10A 250V, glass tube
F3	Terminal near transformer	8A 250V, glass tube
F3, F4	Power board	6A 125V, glass tube
F1	Power board	1A 250V, glass tube

10

Troubleshooting and Error Messages

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This chapter helps the user recover from most of the problems that occur while operating the Headspace Sampler.

Table of Errors

Table 24 lists various errors that can occur. Go to the page shown on the right side of the table for instructions to treat the problem. Diagnostic error messages can have values of OK, LOCK, or FAIL. FAIL errors point to the component that requires troubleshooting.

Table 24 List of errors

Type of symptom	Observation/Error message	Page
Chromatographic results	No peaks	168
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	Poor retention time reproducibility	169
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Vial handling	Vial not dropped	173
	Remove all vials	173
	210 Tray too slow error	173
	211 Tray motor encoder error	173
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Table 24 List of errors (continued)

Type of symptom	Observation/Error message	Page
Heated zone temperature	Temperature actual reads “open”	178
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	Zone at or near ambient	178
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	Analysis aborted Oven Temp. Error	178
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	540 Serial I/O error	182
	550 Serial I/O error	182
	560 Serial I/O error	182

Table 24 List of errors (continued)

Type of symptom	Observation/Error message	Page
Memory and processor errors	600-605 Memory errors	183
Blank display	Display lights up, no self-test at power on, fans running	184
Instrument dead	Power on sampler, no response	185
Lost method in active memory	Loaded new method or chained methods and lost the method in active memory	185

Chromatographic Results

No peaks or reduced sensitivity

There are no peaks when the sample does not reach the GC detector. Check the following general areas for problems:

The GC

First verify that the GC works properly. If possible, mount an automatic liquid sampler (ALS) over the inlet and inject a sequence of internal or calibration standard directly into the inlet. If peaks appear then the problem is not in the GC.

The Headspace Sampler gas source

The gas source may be low on gas or set to the wrong pressure. Check the following gas settings:

- Use 60 to 80 psi for MPC carrier and vial pressurization gas.
- Use a pressure above the pressure inside the sample vial after equilibration for EPC vial pressurization gas (usually 10 to 20 psi).
- Use the inlet pressure setting for EPC carrier gas.

The sample vial

Check for the sample vial for cracks or a defective seal.

The internal Headspace Sampler flow paths

Check the following areas of the Headspace Sampler flow paths:

- The source gas for vial pressurization is connected to the correct bulkhead fittings. For example, do not connect the vial pressurization gas to the MPC inlet when running the vial pressurization gas in EPC mode.
- The source gas for carrier gas is connected to the correct bulkhead fittings. For example, do not connect the carrier gas to the MPC inlet when running the carrier gas in EPC mode. In GCs with EPC, a lack of flow from the Headspace Sampler to inlet usually causes an inlet shutdown error.

- The pneumatic toggle switches for vial pressurization and carrier gas are set correctly. For example, set the carrier gas toggle switch to EPC when using the Headspace Sampler in EPC carrier gas mode. See Pneumatics on page 32. In GCs with EPC, a lack of flow from the headspace to inlet usually causes an inlet shutdown error.
- Verify there is a flow of carrier gas from the Headspace Sampler to the GC inlet. For MPC, the carrier pressure is set so that the sampler provides most of the flow to the inlet.
- Check for restrictions that block the flow path for filling the sample loop. The flow path starts at the sample probe and runs to the bulkhead fitting. See Leak Test on page 148.
- Check for leaks in the carrier flow path. The flow path starts at the sample probe and ends at the GC inlet. See Leak Test on page 148.

Poor retention time reproducibility

Poor retention time reproducibility results from changes in the time it takes the sample to reach the GC detector. Check the GC and Headspace Sampler flow paths.

Check for leaks and restrictions in the carrier flow path. The flow path starts at the sample probe and ends at the GC inlet. See Leak Test on page 148.

Poor area count reproducibility

Poor area count reproducibility results from changes in the amount of sample reaching the GC detector. Check these general areas:

The sample vial

Check the vials that contain the samples that show low area counts. Look for cracks, defective seals, and poor crimps.

The GC

- Verify that the GC works properly. If possible, mount an ALS over the inlet and inject a sequence of internal or calibration standard directly into the inlet. Check the area count reproducibility.
- If Check for Ready is set to “5890 Wait” or “APG Wait” vial equilibration times may vary. Increase the GC Cycle Time or select a different Check for Ready setting.

The equilibration of the sample vial

- Poor choice of equilibration temperature or time. For the best reproducibility, the analytes in the sample and in the headspace of the vial must reach static equilibrium. Increase the equilibration time (see page 55) and/or increase the oven heated zone temperature (see page 52).
- If the sample is a liquid, try adding shake to speed up equilibration. See page 57.

Sample preparation

Check for inconsistent sample preparation technique and vial cap crimping.

Carryover in air or solvent blanks

In theory a run containing a blank made after a run with a sample has no peaks in the chromatogram except for the analytes from the lab air or solvent that is trapped in the sample vial. Carryover results when sample condenses on the flow path or is trapped in unswept area from a sample injection. Run a blank to see small quantities of these analytes.

If the amount of carryover in a series of solvent blanks decreases by a factor of 5 to 10 for each run, the carryover is probably due to an unswept area. Check the following:

- There is flow from the GC inlet to sweep the inlet weldment.
- Check the transfer line connection to the GC inlet.

If the amount of carryover in a series of solvent blanks does not decay for each run, the carryover is probably due to analytes adsorbed or condensed on the flow path or in the inlet. Check the following:

- Poor choice of heated zone temperature settings. See page 52.
- Poor choice of vial pressurization pressure or time. See page 57.
- Flow path or GC inlet contamination.
- Poor choice of inlet.

Unwanted background noise or peaks

In theory a run containing a sample has no unrecognized peaks in the chromatogram. Unwanted peaks or ghost peaks result when an unknown is present in the sample vial or flow path.

Verify that the GC works properly. If possible, mount an ALS over the inlet and inject a sequence of internal or calibration standard directly into the inlet. If there are no unrecognized peaks, the problem is not in the GC.

Check the following in the Headspace Sampler:

- Possible contamination in sample preparation materials and techniques.
- Check for leaks and restrictions in the carrier flow path. The flow path starts at the sample probe and ends at the GC inlet. See Leak Test on page 148.
- Possible contamination in lab air. Run a standard after purging the vial headspace with nitrogen or argon.

Vial Handling

Vial not dropped

This error results when a vial fails to lower from the tray to the carousel. A sensor checks for a vial after the tray lifter lowers the vial. Check the following then call an Agilent service representative:

- Tray belt tension. See page 139.
- Tray position 1 alignment. See page 140.
- Carousel alignment. See page 142.
- Obstructions or dirt

See Recovering from a vial delivery error on page 177.

Remove all vials

This error appears when the sampler tries to recover vials after a vial handling error or after pressing the stop button. The message indicates that the sampler found vials in an unexpected carousel location. See Recovering from a vial delivery error on page 177.

210 Tray too slow error

This error results when the tray motor encoder senses an obstruction. Check and clear any obstruction to the tray chain. See Recovering from a vial delivery error on page 177. Call an Agilent service representative with the number and name of the error.

211 Tray motor encoder error

This error results when the tray motor encoder is not responding. See Recovering from a vial delivery error on page 177. Call an Agilent service representative with the number and name of the error.

212 Tray position 1 error

This error results when the tray position 1 sensor is not found after several attempts.

- Check the tray chain tension. See page 139.
- Check tray position 1 alignment. See page 140.

See Recovering from a vial delivery error on page 177. Call an Agilent service representative with the number and name of the error.

220 Shutter error

This error results when the shutter stepper motor senses an obstruction or cannot find its home position. Check for obstructions in the shutter area and check the tray shutter group alignment. See Recovering from a vial delivery error on page 177. Call an Agilent service representative with the number and name of the error.

230 Tray lifter error

This error results when the tray lifter motor encoder senses an obstruction or cannot find its home position.

- Check and clear any obstruction to the lifter
- Check the carousel alignment. See page 142.

See Recovering from a vial delivery error on page 177. Call an Agilent service representative with the number and name of the error.

240 Sample probe lifter error

This error results when the sampler lifter motor encoder senses an obstruction or cannot find its home position.

- Check and clear any obstruction to the lifter.
- Make sure vial size setting is correct. See Advanced Function 12 on page 80.
- Check the carousel alignment. See page 142.

See Recovering from a vial delivery error on page 177. Call an Agilent service representative with the number and name of the error.

250 Carousel error

This error results when the carousel motor encoder senses an obstruction or cannot find its position.

- Check and clear any obstruction to the carousel.
- Check the carousel alignment. See page 142.

See Recovering from a vial delivery error on page 177. Call an Agilent service representative with the number and name of the error.

260 6-port valve error

This error results when the 6-port valve motor encoder senses an obstruction or cannot find its home position.

- Check and clear any obstruction to the valve motor linkage.
- Check the for any rubbing on the back side of the insulated cover to the loop heated zone.

See Recovering from a vial delivery error on page 177. Call an Agilent service representative with the number and name of the error.

Vial not found in oven

This error appears when the sampler tries to recover vials after a vial handling error or after pressing the stop button. The message indicates the sampler did not find a vial in the carousel where it expected to find the vial. See Recovering from a vial delivery error on page 177.

Tray full!

This error appears when the sampler tries to recover vials after a vial handling error or after pressing the stop button. The message indicates that the tray is full and it cannot check for vials in the carousel. Carefully remove one vial from the tray and reset the Headspace Sampler with Advanced Function 10 (see page 78) or power cycle the instrument. See Recovering from a vial delivery error on page 177.

Recovering from a vial delivery error

Perform these steps to recover from a vial delivery error:

WARNING

The surfaces of the oven, carousel, and oven lid may be hot and could burn you. Before beginning, set the oven and loop temperatures off. Allow the oven to cool to room temperature. Note that vials may remain hot even if the oven has cooled.

1. Raise the lid of the Headspace Sampler.
2. Remove the vial that caused the vial delivery error.
3. If desired, manually remove any vials remaining in the oven. Manually removing the vials is much quicker than allowing the Headspace Sampler to automatically remove the vials, but vials may be hot or hard to reach.
4. Investigate possible causes for the vial delivery error. If something is out of alignment the Headspace Sampler automatic vial recovery routine may cause the same error a second time. See Checking Alignments on page 139.
5. Reset the Headspace Sampler with Advanced Function 10 (see page 78) or power cycle the instrument.
6. The Headspace Sampler asks if it should attempt to automatically recover vials from the oven. If the vials were manually removed in step 3, press **Clear** to deny the automatic vial recovery. Also press **Clear** if there are alignment errors that were not resolved in step 4.

Heated Zone Temperatures

Open or short

Heated zone temperature errors can result from electrical problems or insulation problems.

If the actual temperature reads “open” or “short”, the temperature sensor for that zone is disconnected or damaged. Call an Agilent service representative.

Zone at or near ambient

If the actual temperature is near ambient, check the following:

- Zone setpoint is “0” or OFF. See page 52.
- The fuse for the heated zone is blown. Call an Agilent service representative.

Zone does not stabilize

If the actual oven temperature is slow to respond or does not reach the setpoint, check the following:

- The oven lid is closed and the latches locked.
- The loop heated zone cover sits flush with the base of the pneumatic compartment.
- The line voltage selector is set correctly. For example, if the voltage at the electrical outlet is from 90 to 132 VAC, the line voltage selector switch is set to 115 VAC.

Analysis aborted due to temperature error

During a run if the actual temperature is outside the range of ±4 °C the instrument aborts the sequence and posts an error message. If this occurs, check the following:

- The oven lid is closed and the latches locked.
- The loop heated zone cover sits flush with the base of the pneumatic compartment.
- Call an Agilent service representative.

Pressure Readings

GC goes not ready during a run

When using the AUX EPC to pressurize the vial it is normal for the GC to go not ready for 10 to 20 seconds during the sample extraction. The tubing from the AUX EPC module to the shutoff valve is 1/16-inch OD tubing. The tubing from the shutoff valve is about 6 inches of 1/8-inch OD copper tubing. If the 1/8-inch tubing is longer, it can cause a problem.

GC pressure reading does not match Headspace Sampler pressure reading

Pressure readings at the Headspace Sampler vary slightly from GC pressure readings. The instruments use a different type of sensor and the two sensors are calibrated using a different process. If there are severe differences in the pressure check for leaks and restrictions. See Leak Test on page 148.

Pressures are below normal operating pressures

If pressures are below normal operating pressures, check the following:

- Check the pressure of the gas source (tank, generator, or manifold). For example, the tank may be low on pressure.
- Make sure the shutoff valves on the back of the Headspace Sampler are on.
- Check for leaks between the source and the bulkhead fittings of the GC (EPC) or Headspace Sampler (MPC).
- Check for large restrictions or leaks in the Headspace Sampler. See Leak Test on page 148.

Synchronization With GC Start or GC Ready: GC Does Not Start

If the Headspace Sampler does not start the GC, check the following:

- The Remote Start/Stop cable connection
- The Check for Ready setting, Advanced Function 9

Communication Between the PC and Headspace Sampler

This type of problem results from a break in the communication between the computer and the Headspace Sampler. Check the cable connections between the computer and the sampler.

Check the following for Headspace Samplers set up with a LAN connection:

- The Headspace Sampler and computer have compatible network addresses. For example, the IP address are compatible on the same network.
- The Enable RS-232 setting is OFF. See Advanced Function 16 on page 83.
- The LAN address is correct. See Advanced Function 14 on page 82. Reset or power cycle the Headspace Sampler for IP address changes to take effect.
- Use the ping command from a PC command window to test the connection.

Check the following for a Headspace Sampler with an RS-232 connection:

- The com port settings are correct. Bits per second: 9600; Data bits: 8; Parity: None; Stop bits: 1; Flow control: Hardware.
- The Enable RS-232 setting is ON. See Advanced Function 16 on page 83.

Internal Communication Serial I/O Errors

These errors indicate a bad connection between the main processor PCA and peripheral PCAs. Call an Agilent service representative.

Memory and Processor Errors

These errors result from defects in firmware or a defective main processor PCA. Call an Agilent service representative.

Blank Display

A blank display results from an interrupted or incomplete firmware update. The instrument fans are running, there is no self-test, and the display is lit but blank. Call an Agilent service representative to run the “Recover from Blue Screen” procedure.

Power ON, No Response

This symptom results when the line voltage from the wall outlet does not reach the internal power supplies of the instrument. Check the following:

- Headspace Sampler line module fuses are open or blown.
- Check the power selector switch setting. Fuses will blow if switch is set to 115 VAC and the line voltage is 220-260 VAC.
- Power cord not connected.
- Power ON switch, set to OFF.
- Line voltage not available at electrical outlet.

Lost Method in Active Memory

Methods in active memory are not saved when loading a new method or chaining methods. There is no warning message before overwriting the method in active memory. This is not an error. Be sure that the method in active memory is stored in one of the four storage locations before loading or chaining a new method to avoid losing it. See page 60.

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